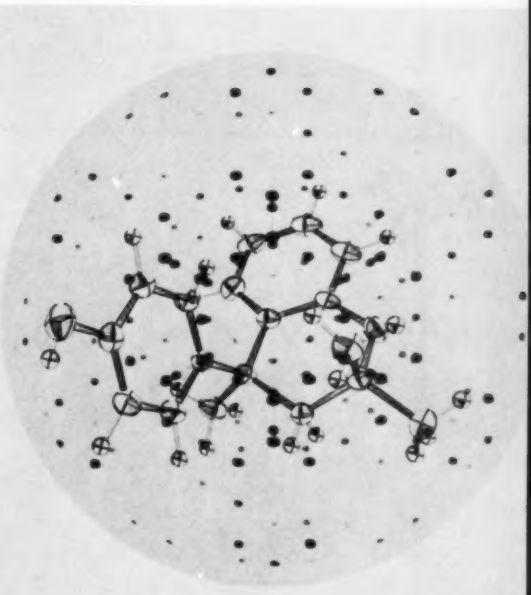
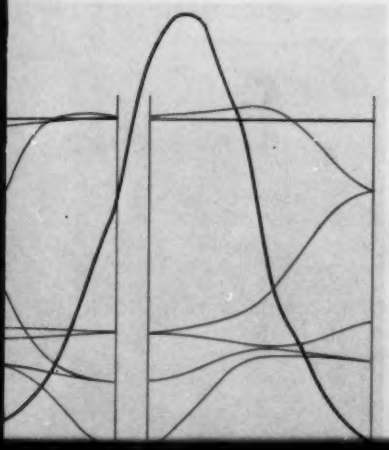
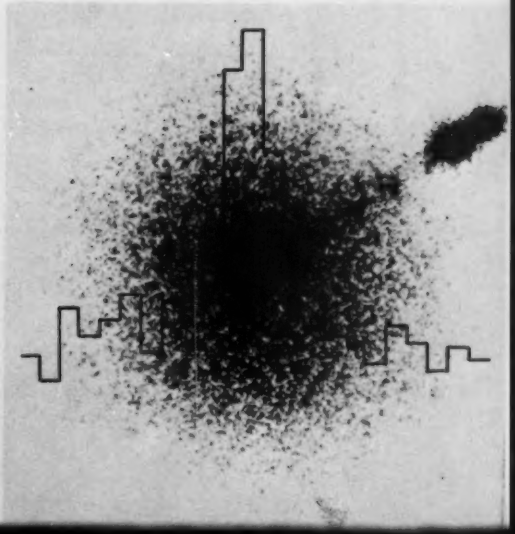
NAVAL RESEARCH

REVIEWS

NOVEMBER 1970







Portraits in Science

Dr. William Lipscomb is noted for developing low-temperature x-ray diffraction methods for the study of single crystals which enabled him to establish most of the presently known chemical structures of electron deficient compounds. His work led also to the general recognition of polyhedral structures. These researches were supported by the ONR Chemistry Program from their inception. Recently he established the structure and stereochemistry of leureoristine which is used in leukemia therapy. After receiving his Ph. D. from California Institute of Technology, Dr. Lipscomb taught at the University of Minnesota and Harvard where he is Professor of Chemistry today.

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Navy X-Ray Research and Utilization

David J. Nagel
Nuclear Physics Division
Naval Research Laboratory

November 8, 1970 marks the 75th anniversary of the discovery of x-rays by Wilhelm Roentgen. That event and subsequent research have filled out a region of the electromagnetic spectrum which has great basic and practical value. As indicated in Figure 1, the boundaries between the x-ray and neighboring regions are not sharply defined. However, the x-ray region can be said to run from 0.1 to 100 angstroms (10^{-8} cm) in wavelength, that is, from about 0.12 to 120 keV in photon energy.

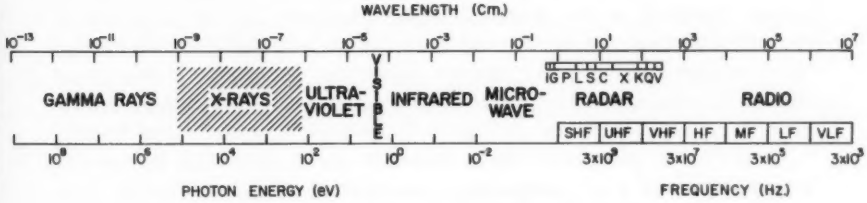

Figure 1 — The electromagnetic spectrum

Roentgen's discovery had immediate and strong impact, both technologically and socially. The situation in the years right after 1895 was similar to what we have witnessed more recently with the advent of transistors and lasers. Work in the x-ray field has remained vigorous. At present, there is continuing development of new x-ray techniques as well as ever-increasing use of x-ray methods developed earlier.

The Navy is both a leading developer and user of methods in the x-ray region. The heavy use of x-rays by the Navy is not dissimilar from that of the other services and many other government and civilian organizations. However, the breadth and depth of the expertise which the Navy possesses for x-ray research tends to be unique. This is attested to by the fact that much of the story of progress in the x-ray field during the past quarter-century is an account of Navy work. The research advances continue to benefit the Navy and other organizations.

This article contains highlights of research progress in developing x-ray techniques plus a discussion of some of the many uses of x-rays. Attention and reference are given mainly to Navy work, particularly

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recent advances and current studies. It is not possible in an article of this length to discuss in detail the relation of Navy x-ray research to other work in the field.

The x-rays areas covered here include: radiography, radiation damage and dosimetry, diffraction, and spectroscopy of cold and hot sources. These are presented in the order of their development although this article is not a complete history even of Navy x-ray work. In general, x-rays are used to obtain information on matter in any of its states, solid, liquid, gas, or plasma, or to alter the properties of materials. X-ray research and applications accordingly require knowledge of sources of x-radiation, and x-ray propagation and absorption. Before considering each x-ray area in detail, it is useful to consider the highlights of how x-rays are generated and interact with matter.

X-Ray Generation

Photons with energy in the x-ray range are created during radiative transitions or arise from conversion of photons in neighboring energy ranges. Involved in the photon creation processes are electrons which can suffer an energy loss in going from one state to another, this energy being radiated as an x-ray photon. Electrons also suffer a change of energy in conversion processes. The various basic interactions which yield x-rays are sketched in Figure 2. Each will be discussed briefly.

Vacancies in electronic states bound to the nucleus can be created by the impact of x- or gamma-rays, electrons, protons, and heavier ions and by nuclear radioactive decay. If the vacancy is filled by an electron from another bound level, the x-ray has an energy equal to the difference in total energy of the two levels and is characteristic of the emitting atom. Intense x-ray line spectra, from any state of matter, arise in this manner. In plasmas, electrons in various unbound (free) states can fill vacancies in bound states, giving x-rays with a range of energies.

High-energy charged particles which experience an acceleration also generate continuous x-ray spectra. In this case, both initial and final states of the radiating particle are unbound, with the emitted x-ray energy equal to the difference in kinetic energy between states. The radiation which results when a high energy electron collides with an electronic field, such as that of the nucleus, is electronic x-ray bremsstrahlung. Similar collision with magnetic fields yields synchrotron radiation, that is, magnetic bremsstrahlung. In another process, the passage of an ultra-high energy (GeV) electron or positron across an interface, for instance from a vacuum into a metal, leads to interface x-radiation.

Conversion of other photons to x-ray photons occurs during Compton scattering. When a gamma ray strikes an electron its energy is divided between the electron and an outgoing x-ray photon in the classical Compton process. Conversely, a high energy electron can lose some of

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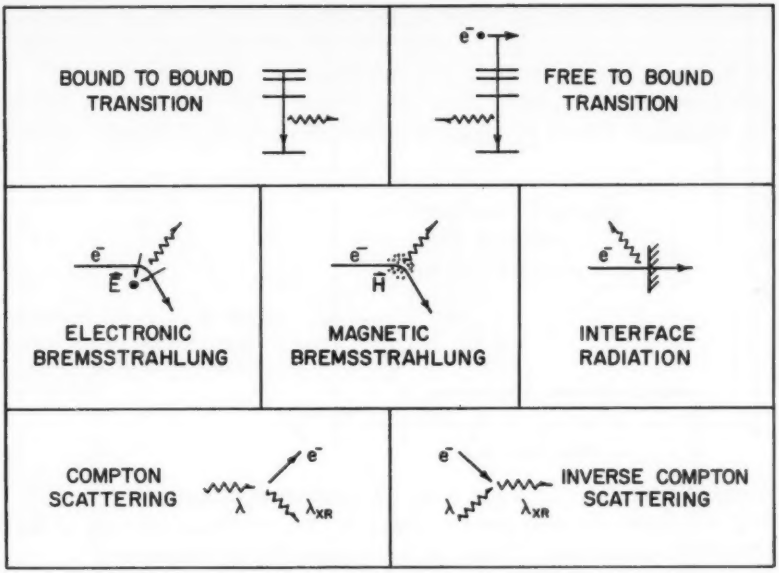

Figure 2 — X-ray generation processes: photon creation by electron transitions to bound states (top row) and between free states (center row), and photon conversion during inelastic scattering (bottom row).

its energy in changing an optical or ultraviolet (UV) photon to an x-ray photon.

These basic physical processes which yield x-rays often occur together, depending on the method of excitation and conditions in the emitting material. Atomic spacing in the source influences the transport of exciting radiation through the emitting region and determines whether the resultant x-rays will exhibit bonding or collision effects. The source density and thermal state also determine absorption as the generated x-rays leave the emitting material. If an x-ray source is optically thin, the measured spectra will evidence only the basic emission processes. In contrast to this, characteristics of the many emission processes active in hot, optically thick sources are washed out due to multiple interactions. Solids offer an intermediate case where the generated x-ray spectra suffer some absorption, yet still clearly reflect the basic processes. Figure 3 shows, for example, the spectrum of an x-ray tube (1). Both characteristic lines and the bremsstrahlung continuum appear in this spectrum, but jumps in continuum emission due to varying self absorption are evident.

Of the various types of x-ray emission, characteristic lines are most useful in determining the makeup and conditions of a source and for the

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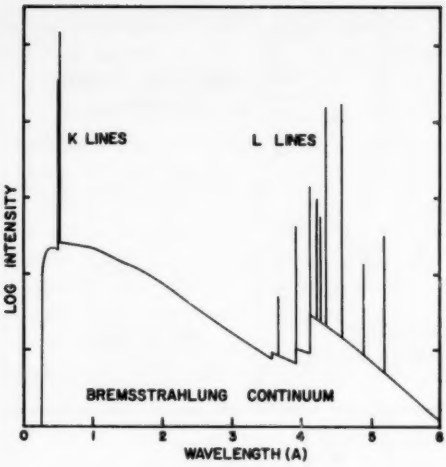

Figure 3 — X-ray spectrum from a rhodium target x-ray tube operated at 45 kev (1)

study of materials. Continua from electronic and magnetic bremsstrahlung processes are also used to study source conditions as well as for absorption measurements and for calibration of x-ray equipment.

Interaction of X-Rays with Matter

We have just seen that, for x-ray generation, there are a few basic processes which along with source conditions determine an emitted x-ray spectrum. The situation is similar for the interaction of x-rays with with matter. Here the processes of interest are x-ray scattering and absorption. These, together with characteristics of the irradiated material, determine the flow (transport) and absorption (deposition) of an x-ray flux.

Elastic, coherent scattering involves no change in the x-ray energy. Such scattering is caused by the electrons in the target atoms. It occurs whether the atoms are alone as in a gas or in a condensed phase. If the target atoms have a regular arrangement, however, x-ray scattering becomes especially strong in some directions. This effect, called diffraction, is as we will see, very useful for determining the atomic structure of matter.

Inelastic scattering occurs during the Compton process. While elastic scattering is sensitive to the arrangement of target electrons, Compton scattering reflects their velocities. Hence, inelastic scattering is used for determining momentum distributions of, for instance, the valence electrons in solids.

X-ray photons are destroyed in the photoelectric absorption effect. In this process, an atomic electron is excited from its ground state level, thus taking up the energy of the absorbed x-ray. The vacancy created

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can lead to further x-ray production termed fluorescence, but any new photon is distinct from the one absorbed.

The probability for the scattering and absorption processes can be given in terms of cross sections or, equivalently, absorption coefficients. Examples for two elements are shown in Figure 4. Except for

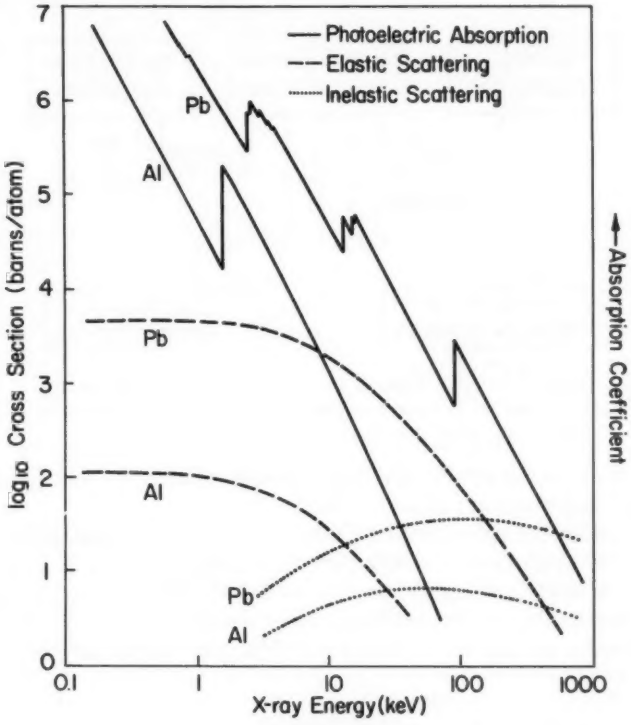

Figure 4 — Cross sections for the interaction of x-ray photons with Al and Pb

the lightest elements, the photoeffect dominates scattering over most of the x-ray range. Jumps in the absorption coefficient occur at edges where x-rays have enough energy to remove electrons from the next most tightly bound atomic shell. The photoelectric effect, with its large and regularly varying absorption jumps, is the hallmark of the x-ray region.

When an x-ray beam shines on a material, the photons which interact will mostly be absorbed photoelectrically, giving excited target atoms and photoelectrons. Fluorescent x-rays and Auger electrons will also result. The kinetic energy of photo and Auger electrons is dissipated

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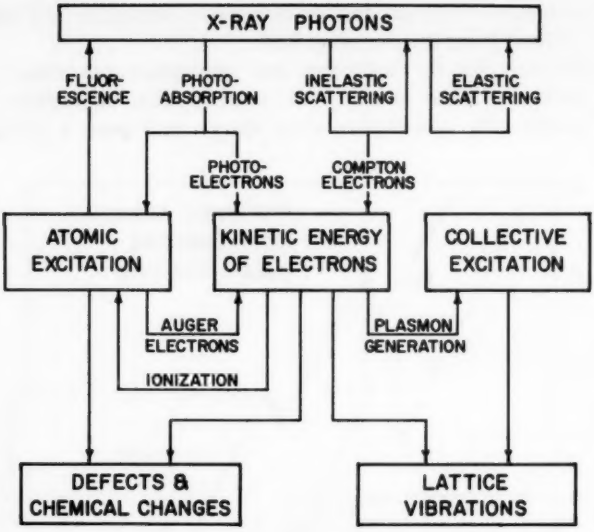

Figure 5 – Flow diagram for the conversion of x-ray photon energy first to electronic excitations and then to defects, chemical changes, and lattice vibrations

quite locally in the lattice. The major processes involved in x-ray transport and deposition are shown in Figure 5.

This outline of the physics of x-ray generation and interaction is all that can be given here. Examples of results obtained in the several areas of x-ray research and application will be shown and discussed in the rest of this article. Generalized schematics of the equipment used to obtain these results are shown in Figure 6 for each of the major x-ray areas.

Radiography

In Figure 4 it can be seen that x-rays have low absorption coefficients relative to UV and that the coefficients are more highly dependent on atomic number for x-rays than gamma rays. These factors combine to allow high contrast radiography of thick specimens. Radiography was the earliest and is the most widely known application of x-rays. X-radiographic techniques extend from those for measurement of massive samples such as ship plate to microradiographic methods which can reveal detail on the order of 10^{-3} cm.

The Navy has been active in advancing radiography techniques. The Non-Destructive Analysis group at the Naval Ordnance Laboratory (NOL) has developed and used several high energy x- and gamma-ray methods mainly for ordnance inspection. In its 25 year history, this group, now headed by Mr. E. L. Criscuolo, has investigated isotope

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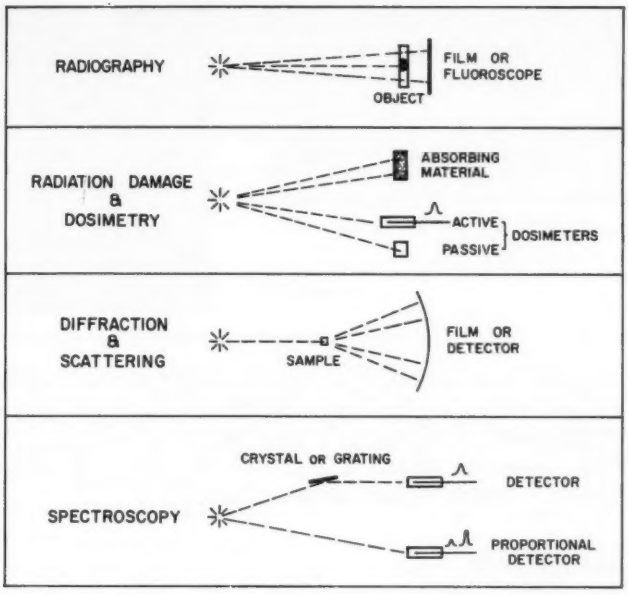

Figure 6 — Schematics of the x-ray source and equipment used for measurements in each of the major x-ray areas

source applications and developed methods for recording radiographs. These include an electrostatic imaging device and a high resolution, high sensitivity fluoroscope. The NOL group uses radiography for inspection of detonators and solid propellant rocket motors, as well as the study of castings and other materials.

Despite the more recent appearance of neutron radiography and other non-destructive testing methods, x-radiography is increasingly used for medical diagnosis and for flaw detection in materials. Thousands of radiographs are taken daily for protection of the health of naval personnel, for production and testing of Navy hardware, and as part of naval research projects.

Figure 7 gives a recent example of conventional macroscopic x-radiography. Teredos are marine borers which enter submersed wood, such as pier pilings, through small holes when they are young. They grow rapidly, eating and moving through the wood. Since they do not emerge again, the wood will be ostensibly sound despite its weakened condition. Means to combat Teredos are under study by Dr. J. D. Bultman at the Naval Research Laboratory (NRL).

The x-ray exposure in Figure 7 was taken with a steady state x-ray source. Due largely to early work at NOL, it is also possible now to make x-radiographs using flash sources with exposure times down to

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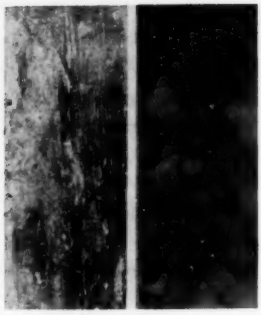

Figure 7 — Photograph (left) and x-radiograph (right) of a piece of apparently sound wood which has been hollowed out by Teredo marine borers

3×10^{-9} seconds. Such machines are used for single shot and sequential photography of ballistic impact and explosive phenomenon. Figure 8 shows the hypervelocity impact of a steel rod on a steel plate. Such work at NRL, under the direction of Mr. W. W. Atkins, is related to the protection of satellites against meteoroid impact.

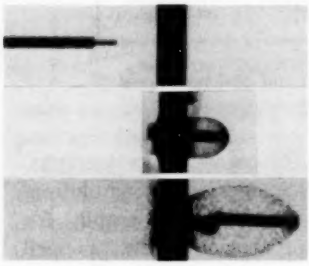

Figure 8 — Sequential flash x-ray photographs of a steel rod moving at 15,000 ft sec and penetrating a 1/4 in. steel plate (edgeview)

Radiation Damage

X-rays penetrate relatively far into materials, but their absorption when it occurs can cause a great deal of damage in the target. The energy of each x-ray photon is about 1000 times in excess of that required to break atomic bonds and cause defect centers. The excess of free electrons created by deposition of x-ray energy is very transient, disappearing in about a microsecond. Longer lasting defects caused by this deposition are of use in the fields of radiation physics, chemistry, and biology, including genetics, and in cancer therapy. The study of mutations due to x-irradiation was begun immediately following the discovery of x-rays.

Work on color centers in alkali halides at NRL provides an example of the use of x-rays to cause damage in materials. Color centers are the defects produced during irradiation which cause color in previously clear crystals. Dr. I. Schneider has shown that the M centers in NaF

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can be used for information storage, as in a computer memory. With Dr. M. N. Kabler and Mr. M. Marrone, he demonstrated storage densities greater than 10^7 bits per cm^2, high writing efficiency, non-destructive readout and long-term stability (2).

Color center information storage is presently in the research stage. There are areas, however, in which x-radiation damage is already used in manufacturing processes. Production of some chemicals, polymerization, and food and seed preservation are examples.

Dosimetry

Advances in knowledge of the damage produced when x-rays interact with matter are often applied to the detection and measurement of x-rays. Dosimetry may be broadly defined as the science and practice of measuring spectra and intensities of radiation of many kinds, including x-rays. In this light it is most comprehensive, the queen of x-ray fields, proproviding all measurements for studies of materials and x-ray sources. In practice, however, dosimetry is more of a handmaiden, providing the tools for monitoring radiation sources, especially personnel radiation recorders for the preventative aspects of Health Physics. X-ray measurements in other areas do indeed use devices developed or used for dosimetry, but they are not usually thought of as dosimetry.

X-ray detectors and dosimetry devices are based on the transient electronic or more permanent chemical or physical radiation damage induced by absorption of x-ray energy. These include fast gas and solid state counters (*e.g.* proportional, scintillation and p-i-n silicon detectors), and time integrating materials, (*e.g.* film and thermoluminescent dosimeters).

Pioneering studies of the defects produced in optical materials and associated luminescence have been made at NRL by Dr. J. H. Schulman, Dr. C. C. Klick and their colleagues. The luminescence emitted by the decay of radiation-induced defects may be spontaneous or may occur only when the material is heated. The latter case is termed thermoluminescence (TL). TL devices are read out after exposure by measuring the light emitted as the TL material is heated. The intensity of such "glow curves" is directly related to the dose. New TL materials have been discovered at NRL. An example is CaF_2 doped with Mn developed by Mr. R. J. Ginther (3).

The more basic work at NRL has been used by Mr. F. H. Attix in the development of practical dosimeters (4). Glow curves for two different TL materials in a glass envelope at four levels of exposure to Co^{60} gamma rays are shown in Figure 9. When TL material is mounted with heater electrodes in this way, a convenient, easily-read device results. It offers an alternative to film badges for personnel radiation monitoring (5).

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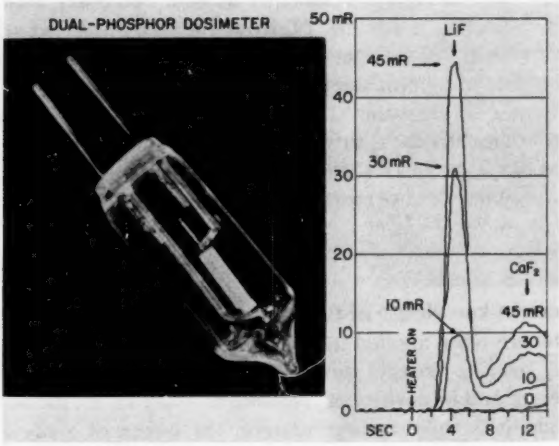

Figure 9 — Glow curves (right) obtained from a bulb-type dosimeter containing LiF and CaF_2 (left) (5). Exposure levels are given in milliroentgens.

Structure Determination Using X-Rays

As indicated in Figure 6, the experimental arrangements used in dosimetry and radiation damage, as well as radiography, are somewhat simpler than those for structure studies by x-ray diffraction and scattering. For structure determination, the x-ray source provides a tool for probing the arrangement of atoms in perfect and imperfect materials. The information obtainable from a variety of x-ray diffraction and scattering measurements is summarized in Table 1.

Most of the diffraction and scattering methods listed in Table 1 have been thoroughly developed and are widely used in studies of metallurgical and biological materials and minerals. For some of these methods, the early advances were made at NRL. As an example, present day powder crystal diffractometers can be traced back to Navy work with geiger counters in the 1940's. Other techniques listed in Table 1 still require vigorous research efforts to determine their ultimate utility. Prime among the x-ray diffraction methods under current development is the rapid determination of the number and arrangement of the various atoms in large organic molecules. Such work has been pursued for several years and is still flourishing at NRL.

X-ray diffraction was discovered by von Laue and his students in 1912. Thousands of structure determinations have been made since then. These range from the simplest inorganic salts done before 1920 to complex proteins. In order to determine a complicated crystal structure using x-rays, the amplitude and phase of the diffracted waves must be known. But phase information is unavoidably lost in the process of

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TABLE 1
Information Obtained from X-Ray Diffraction and Scattering Measurements of Perfect and Defect Materials

Structural Perfection of Sample	X-Ray Measurement	Information Obtained
A. Perfect	Direction and intensity of diffracted x-ray beams	Single crystal orientation, chemical identity, structure and electron distribution.
		Chemical identity and structure of crystalline powders.
B. Zero and one dimensional defects	Topograph	Interstitial and vacancy concentration, and dislocation type and arrangement.
	Diffraction profiles	Single crystal substructure and dislocation density.
C. Two dimensional defects		
Internal		
Stacking faults	Diffuse scattering	Stacking fault probability
Grain boundaries	Topograph and texture measurements	Grain size and orientation
External		
Surfaces	Line broadening and small angle scattering	Crystallite size
D. Three dimensional defects		
Crystals	Diffuse scattering and diffraction profiles	Short range ordering and clustering, and static displacements (atom size, strain).
	Diffuse scattering and diffraction intensities	Dynamic displacements (phonon spectra and vibration amplitudes).
Amorphous material	Scattering	Radial distributions

measuring diffracted x-ray intensity. This led to the development of various indirect methods of structure determination. Such approaches are non-routine and require long periods of expert chemical and interpretative work. One structure determination commonly constituted a PhD thesis.

In the late 1940's it was realized that the structure problem was overdetermined if atomic scattering factors are used. Diffraction experiments yield about 25 data points for each unknown crystal coordinate. This led Dr. J. Karle and Dr. H. A. Hauptman at NRL to

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prove in 1950 that a direct solution exists for the phase determination problem (6). The fact that calculated electron densities must turn out to be positive led to the development of inequalities which the phase angles must satisfy. These inequalities and many subsequent theoretical developments at NRL have made possible direct methods of phase determination (7).

The theory was implemented in a practical system, the Symbolic Addition Procedure, by I. L. and J. Karle in the mid-1960's (8). This method involves the use of symbols for a few of the unknown phases during a structure determination. It has been tested on compounds of increasing complexity and difficulty. Now it is possible to routinely and automatically determine the structure of crystals containing a center of symmetry. Structures of several non-centrosymmetric crystals with up to about 250 atoms of nearly equal scattering power in the unit cell have also been determined using the Symbolic Addition Procedure. Karles' method has proven to be a fast tool for structure determination which requires only a small (~10 microgram) amount of material. The chemical formula of the material under study need not be known, a fact important for the structure determination of the photo rearrangement products obtained when organic compounds are irradiated with ultraviolet light. Vibrational amplitudes are obtained in addition to atomic positions during a structure analysis.

The structures determined by the Karles and their collaborators in the course of developing procedures have been of major importance in a wide variety of studies in addition to UV radiation damage (9). The medically important materials whose structures were determined at NRL include anti-radiation and antibiotic compounds; drugs for treatment of hypertension, nervous disorders and for heart therapy; and some complex poisons. The structure of reserpine is shown in Figure 10. This compound is now the commonly used drug for treating high blood pressure (10).

Structures of the many other biologically important compounds have been determined using the Karles' method. Adenosine triphosphate is a prominent example (11). This compound plays an essential part in the metabolism of all living organisms, being the key molecule for energy transfer. Figure 11 shows its structure, the largest non-centrosymmetric crystal analyzed to date using the Symbolic Addition Procedure. It was found by a group at Cambridge, England. To quote from the Cambridge article, "Recent developments in the direct solution of light-atom structures, particularly by Karle and Karle, have opened up the possibility of obtaining structural information about complex, asymmetric molecules even from very limited data."

Dr. Hauptman has pursued a direct method of structure determination which is distinct from the Symbolic Addition Procedure. Some important formulas can be used in both approaches, but Hauptman's method does

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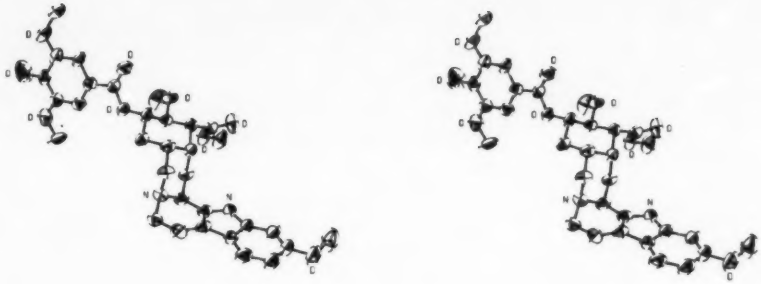

Figure 10 — Sterographic drawing of reserpine (10). With some practice, this can be seen in 3D without a viewer if the drawing is held 1 to 2 feet from the eyes.

not involve the use of symbols. Instead, a two-step procedure is used in which so-called "cosine invariants" are first calculated from the diffraction data. These quantities are then used to compute the phase angles and atomic arrangement. Computer programs for both steps have

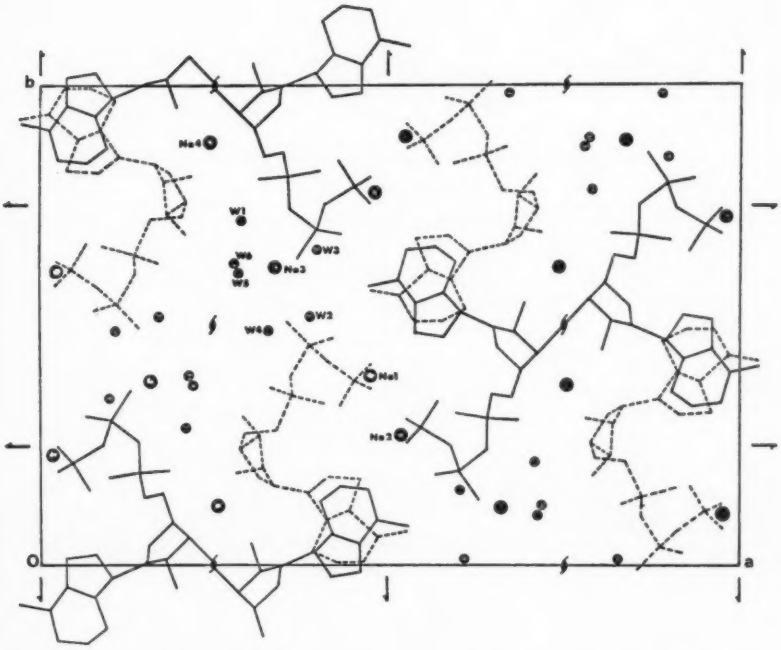

Figure 11 — Packing diagram for a sodium salt of adenosine triphosphate (11). Solid and dashed lines connect the atoms in molecules with two distinct geometries. Sodium atoms, associated water molecules and symmetry symbols are also shown.

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recently been written (12). To date, this method has yielded the structures of several non-centrosymmetric crystals, including one with over 100 nearly-equal atoms in the unit cell (13).

In most of the other diffraction-related Navy work, existing techniques are being used in the course of research projects on metallurgical and inorganic materials of practical interest. This is the case for x-ray techniques aimed at perfect crystal structure determination as well as defect crystal methods. Examples in both areas, especially the study of transformations, will be given in the rest of this section.

Dr. F. E. Wang at NOL has used x-ray diffraction to unravel several highly complex crystal structures in the intermetallic titanium-nickel alloy TiNi (14). This material undergoes a near-room temperature martensitic transformation, giving it a "structural memory" which is useful for switching applications. Nonmagnetic, ordnance-disposal tools are also made from TiNi. Knowledge of the TiNi structure gained from diffraction experiments is useful in these applications and also provides a guide in searching for other compounds to meet specific needs. To date, intermetallic compounds have proven broadly useful, their applications ranging from the age hardening of aluminum alloys to high transition temperature superconducting devices.

Also at NOL, Dr. J. R. Holden and Mr. C. Dickinson use x-ray diffraction techniques to determine the structure of nitro compounds which are explosives (15). They also study the intermediate compounds used in making explosive materials. Hence, the work relates molecular structure to both preparation and properties of ordnance materials. Knowledge gained from such comparisons contributes to the development of explosives which have high energy content but are nonetheless easy to prepare, handle, and store.

At NRL, Dr. F. L. Carter is doing structure analysis on materials containing transition and rare earth metals. For example, the structure of K_2MnF_4, a layer-by-layer anti-ferromagnetic material was recently refined. This material is of interest because of its two-dimensional nature and possible use in infrared devices.

We now turn to x-ray methods for measurement of defect structure. Of these, x-ray topography has proven to be a fast way to determine the quality of crystals. In this technique a diffraction photograph of the surface of the test piece reveals areas of structural irregularity. Using topography, Mr. L. S. Birks showed that ruby laser crystals can be screened for the requisite perfection prior to expensive cutting or operational tests (16). X-ray pictures from good and poor quality ruby crystals are shown in Figure 12. Topographs taken more slowly with higher resolution also reveal dislocations, for instance, in the silicon wafers used for integrated circuit manufacture. The use of x-ray topography allows correlation of structural defects and device mal-performance or failure.

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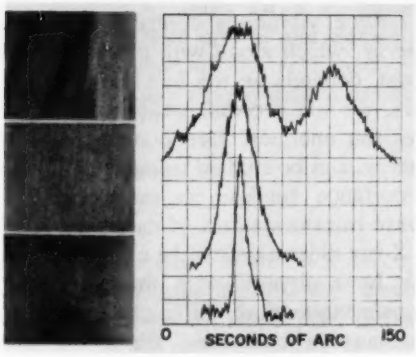

Figure 12 – X-ray topographs and diffraction profiles from synthetic ruby laser crystals of varying structural perfection. Better crystals give featureless topographs and narrow profiles (16).

Topography is a graphic but non-quantitative means of studying defect crystal structure. Diffraction profiles provide more precise information on the perfection of single crystals. Such data from ruby crystals (16) is also shown in Figure 12. More perfect crystals yield narrower profiles because of their lower dislocation density. Crystals with appreciable substructure give broader, and even multiple profiles. The width of the diffracted line can be used to calculate dislocation density. The diffracted intensity can also be interpreted in terms of structure perfection. Theories of diffraction from perfect and imperfect crystals have long been available but an intermediate theory applicable to most real crystals was developed by Zachariasen only three years ago. This is being applied to the interpretation of diffraction intensities by Dr. D. B. Brown at NRL. Diffraction profiles from a wide variety of crystals are being measured by Dr. M. Fatemi and Mr. C. M. Dozier. Work with the width and height of diffraction profiles is providing a means to relate structural perfection to the growth and mechanical-chemical histories of a crystal.

X-ray diffraction has also been used at NRL for measurement of the most universal of three dimensional lattice "defects," namely atomic vibrations. The motion of atoms in solids has sweeping consequences in determining the thermal, chemical, electrical and mechanical properties of materials. Low intensity thermal diffuse x-ray scattering, which occurs between diffraction maxima, can yield detailed information on the spectrum of lattice vibration frequencies. Mean square amplitudes of atomic vibration can be derived from the intensity of the diffraction peaks themselves. An analytical method for evaluating thermal scattering contributions to the diffraction maxima was developed by E. F. Skelton and J. L. Katz (17). Dr. Skelton has used diffraction intensities to determine atomic vibration amplitudes in two hexagonal structures, those of zinc and holmium (18).

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Lattice vibrations contribute to the entropy of a system. Internal energy, which along with entropy determines the free energy in a material, depends on the bonding electron energies. In a subsequent section, we will see that x-ray spectra can be used to determine valence electron energies. Hence both parts of the free energy, entropy and energy, can be studied using x-ray methods. The free energy has central importance because its minimization determines the structures and phase transformations of materials, and hence many of their properties.

X-ray diffraction can be used to probe phase transformations directly. We have already cited the work of Wang on temperature-induced transformations in TiNi. Extensive work on the high temperature austenite to ferrite and bainite transformations in steels was done by Mr. L. S. Birks and Dr. H. Friedman at NRL, starting in the mid 1940's. X-ray diffraction was used as a probe to detect structural changes as a function of temperature and stress (19). At present, Dr. A. W. Webb is using a large tetrahedral press capable of exerting 100,000 atmospheres pressure to study transformations in solid electrolytes. X-ray powder diffractometry provides data on structure and lattice parameter as the pressure is increased. Dr. Skelton is using diffraction in conjunction with a smaller pressure cell to study the pressure dependence of bonding forces in model materials such as NaCl. High pressure studies are important because of their relation to deep submergence and geophysical problems.

Another example of diffraction work which spans three decades at NRL involves quartz oscillator cyrstals which are used as frequency standards. In the early 1940's Friedman devised a method of orienting quartz crystals with x-ray diffraction prior to cutting (20). If a cut crystal did not have the desired frequency, x-rays were used to harden and thus tune it by radiation damage. This pair of x-ray methods solved an old problem and was immediately adopted for wartime use. Now quartz crystals are being used as time standards in Navy navigation satellites. Again radiation damage enters, but now as a problem. It appears that the topography and diffraction profile measurements used by Birks may allow preselection of crystals which will be less susceptible to radiation damage in orbit.

In this section, we have emphasized work aimed at developing diffraction techniques, and materials studies for which x-ray diffraction provides the primary data. In many other projects, x-ray diffraction is just one of several tools used. There is wide-spread, routine use of x-ray diffraction for crystal orientation, stress and chemical analysis and structural characterization in the course of much materials research. For example, at NRL Mrs. J. B. Burbank uses diffraction to measure the lattice spacings and chemical identity of compounds which appear in lead and silver batteries (21). Such work was initially done by Dr. C. S. Barrett at NRL in 1933. This type of research is now related to power supplies for

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submarines and pollution-free electrical automobiles. Dr. J. E. Davey, also at NRL, uses x-ray diffraction, along with optical and electronic means, for the total structural characterization of thin semiconductor films (22). These are made into microwave and electro-optical devices.

X-Ray Spectroscopy

Unlike diffraction studies where the x-ray source is merely a tool, the immediate goal of spectroscopy is the measurement of the spectra (curves of intensity versus photon energy) of various sources themselves. However, the ultimate goal remains knowledge of the condition of atoms and ions in the source. As shown in Figure 6, x-ray spectroscopy can be done in either of two ways. The first requires a crystal or grating for physical disperson of the spectra. In the second, so-called non-dispersive method, use is made of detectors whose output pulse size is proportional to photon energy. Electronic pulse height discrimination is then used to obtain a spectrum. The latter method yields higher intensity but poorer resolution than crystal or grating spectrometers.

The development of crystal spectrometers followed closely the discovery of x-ray diffraction. In 1914, Moseley's spectroscopy measurements showed that the energies of characteristic x-rays follow a simple law. This led to our understanding of what atomic numbers mean. Such work was thus fundamental to our knowledge of the electronic structure of atoms and to the development of quantum mechanics in the 1920's. It also resulted in the discovery of one of the few remaining unknown elements, hafnium, in 1923.

Virtually all kinds of x-ray spectroscopy have been pursued at NRL over the past three decades. The measurement of "cold" atomic and solid state sources was started in the early 1940's and is still being profitably pursued. The work began with spectrochemical analysis using x-ray excitation. Later studies resulted in microanalytical techniques in which x-rays are generated by electron impact. Current x-ray spectrochemical analysis work concerns refinement of x-ray and electron excitation techniques and development of methods using ion excitation. Other solid state spectroscopy at NRL is now aimed at the study of bonding electron structure and its relation to properties. The study of x-rays from hot (plasma) sources began at NRL in the late 1940's with a measurement of solar x-radiation. Spectroscopy of x-rays from more distant cosmic sources and laboratory plasmas was begun subsequently and is still in progress.

Advances in knowledge as a result of spectral measurements of these various sources have resulted from a combination of experiment and theory. However, the capabilities and utility in each area are primarily

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determined by available equipment. The development and improvement of x-ray instrumentation, especially spectrometers, has been largely responsible for the success of work at NRL. Advances have been made in the dispersion and detection of x-rays and in data recording.

In the early 1940's, Friedman and Birks combined high intensity x-ray tubes with geiger counter detectors in a spectrometer design which is still widely used. Such a spectrometer used flat crystals for dispersion (23). In the mid 1950's Birks developed methods for producing curved spectrometer crystals. These find extensive application in high efficiency, concave-crystal, scanning spectrometers (24). More recently Birks has also developed a convex, curved-crystal spectrometer with which an entire x-ray spectrum can be simultaneously recorded (25). In all types of spectrometers, the contradictory traits of high resolution and high efficiency are desired. In his work with spectrometer crystals, Birks and his collaborators have studied means of treating crystals to increase their reflectivity while not degrading resolution too severely. Diffraction profiles, such as those in Figure 12, are used to test flat crystals. Methods to increase the reflectivity of the commonly used crystal, LiF, by a factor of 14 resulted from this work (26). Such an advance translates into reduced measurement times or increased sensitivity for weak x-ray signals.

Non-dispersive x-ray techniques were also furthered by Birks with the application of multichannel analyzers to x-ray spectroscopy (27). Originally developed for nuclear studies, these instruments are now universally used in combination with gas and solid state proportional x-ray detectors. Numerous applications of x-ray spectrochemical analysis, especially the measurement of trace levels, are made possible by this sensitive, rapid combination of x-ray detection and recording equipment.

Many of the developments in laboratory x-ray instrumentation have been applied to flight instrumentation for measurements of x-rays from space. But, it has not been a one-way street. The need for highly-sensitive, remotely-controllable, compact, rugged flight hardware with long-distance readouts has resulted in equipment also of use in the laboratory. For example, in 1963, Dr. R. L. Blake and his colleagues at NRL developed a small, stepping-motor, crystal spectrometer for rocket astronomy (28). The author recently used their design as a prototype to build a spectrometer which conveniently fits into existing vacuum chambers on the NRL accelerators. This is being used for ion excitation studies. Another example of the interplay between flight and laboratory methods concerns thermoluminescent materials. In 1949, $CaSO_4$ TL dosimeters doped with Mn were flown on a rocket by Friedman as compact, time-integrating x-ray detectors. As we already noted, a great deal of other work on TL materials and their laboratory applications has been done at NRL.

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While some x-ray instrumentation is useful in several of the spectroscopy areas, each application of spectroscopy requires separate discussion. In the remainder of this article x-ray spectrochemical analysis using x-ray, electron, and ion excitation, and x-ray spectroscopy for bonding studies will be discussed in turn before considering spectroscopy of laboratory plasmas, the sun, and other sources in space.

As background for the sections on chemical analysis and bonding studies, we should consider the generation of characteristic x-rays in somewhat more detail. Figure 13 shows spectra from Si metal and SiO_2 as an example. Emission accompanies the transition of an electron to

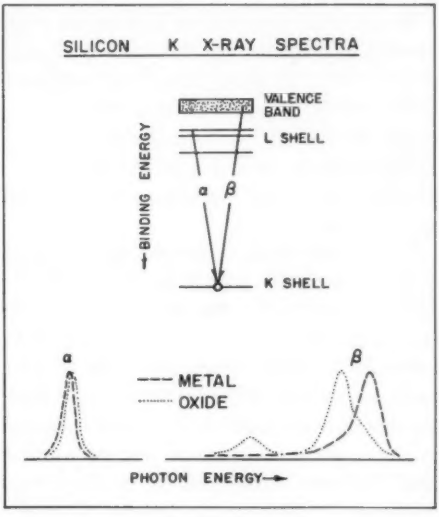

Figure 13 — Energy levels, electron transitions and normalized Si x-ray spectra from pure Si and SiO_2. The Kβ valence band spectrum is strongly dependent on the bonding of the Si atoms.

a hole in an inner shell which is created by x-ray absorption, or electron or ion impact. Electrons from other inner levels yield x-rays which are substantially independent of the chemical state of the emitting atom. It is these harder (higher energy, more penetrating) x-ray lines which have found wide application in elemental chemical analysis. In contrast, transitions of valence electrons to inner shell vacancies give soft x-ray bands which reflect the chemical environment of the emitting atom very strongly. Such spectra accordingly yield information on bonding electron structure.

X-Ray Fluorescence Analysis

Considerable work using characteristic x-ray fluorescence to identify the elements present in unknown samples was done prior to 1940. It

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was not until the mid-1940's, however, that the development at NRL of spectrometers with geiger counter detectors made quantitative x-ray analysis a competitive method. But to obtain highly accurate results, it used to be necessary to compare intensities from the unknown and several standards of known composition. Preparation of the required standards was a nuisance and increased the overall cost of x-ray analysis. The number of standards needed was later reduced by application of mathematical regression methods. Such an introduction of equations with empirically determined parameters has been done in a number of ways. One of them, the Empirical Coefficients method, was developed by Criss and Birks at NRL (29). While optimized for use, this type of approach still ignored the physics of the analysis problem.

In the mid-1950's Sherman at the Philadelphia Naval Shipyard wrote out equations for fluorescence analysis which took into account the physics of x-ray generation and transport (30). These were not practical, however. One problem was the lack of accurately known excitation x-ray spectra. This difficulty was removed by the x-ray tube spectral measurements of Gilfrich and Birks in 1967 (31). Figure 3 shows, for example, an x-ray spectrum more recently measured at NRL by Gilfrich and collaborators (1).

Criss and Birks reformulated a practical Fundamental Parameters method in 1967 which made use of the measured excitation spectra and took secondary fluorescence into account (29). This approach allows automatic, iterative computer calculation of composition using x-ray intensity measurements from the unknown and only one standard for each unknown element. The Empirical Coefficient and Fundamental Parameters methods were tested by analysis of steels (29).

The more mathematical and physical methods for x-ray fluorescence analysis were developed for homogeneous specimens. Many unknowns are indeed either homogeneous to begin with, or are made uniform by various chemical and mechanical procedures. In the latter case, the additional step in sample preparation can be avoided by the development of a suitable and economical method of calculating the x-ray fluorescence from inhomogeneous materials. This would also allow x-ray analysis of specimens for which homogenization is impossible or too expensive. Criss has recently made progress toward a theory for fluorescence from inhomogeneous samples (32) and is extending the analysis to include the complex sample geometrics which occur, for example, in analysis of various phases in alloys.

At present, and largely because of the experimental and theoretical work at NRL, x-ray fluorescence analysis is a broadly applicable, rapid, accurate and low cost method of analysis. The technique is applicable to all elements above beryllium. Levels down to less than one part per million can be measured for heavy elements in light samples, for example, mercury in water and other pollutant analysis problems.

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X-ray fluorescence analysis often requires less than one minute to obtain accuracy of better than 1/2 percent relative for concentrations above about three percent. The cost per analysis is now down to about $1 to $2. These advantages make fluorescence analysis widely used in industrial as well as research applications. Examples include control of steel production and on-stream analysis in chemical and ore-refining plants. The theory and practice of x-ray fluorescence analysis are covered in a book by Birks (33).

Electron Probe Microanalysis

Samples analysed by fluorescence analysis are typically of the order of 1 cm in size. It is possible to use x-ray beams limited to about 1/2 mm in diameter to fluoresce a sample in order to study composition as a function of position on the sample. But, intensity and therefore accuracy are low. While x-rays cannot be focussed to obtain high intensity, electrons are easily formed into beams about 1 μm diameter, making possible x-ray generation and accurate chemical analysis from sample volumes on the order of 10^{-11} cm^3 which contain as little as 10^{-14} grams of the unknown element.

X-ray microanalysis is performed using an electron probe, shown schematically in Figure 14. Such an instrument was originially constructed by Castaing and Guinier in Paris. Birks and Brooks reported the operation of the first microprobe in the U.S. in 1957 (34). An extended research effort followed at NRL, developing new instrumentation for the microprobe, advancing theories relating electron-excited x-ray intensities to composition, demonstrating applications, and studying the interaction of electrons and solids.

The microprobe at NRL was used for work ranging from measuring calcium distributions in bones, through analysis of particulate matter filtered from the ocean, to determining the cause of relay contact failure in some torpedos. Studies of the selective leaching of Cr from 304 stainless steel pipe walls by a liquid lithium heat exchange medium, of grain boundary diffusion, of precipitates in alloys, and of phase diagrams were performed at NRL mainly by Birks, Brooks and Seebold. Most of our present knowledge of interdiffusion in metals and alloys has resulted from the use of electron probes in many laboratories. An example obtained by the author is shown in Figure 15. It includes data taken using both of the major ways to make microprobe measurements, namely a line scan for which the sample was translated under the beam, and an area scan. The x-ray scanning picture resulted from electronically deflecting the electron beam over the sample in synchronism with a cathode ray tube whose intensity was modulated by the x-ray intensity. Many applications of the electron probe are described in a second book by Birks (35).

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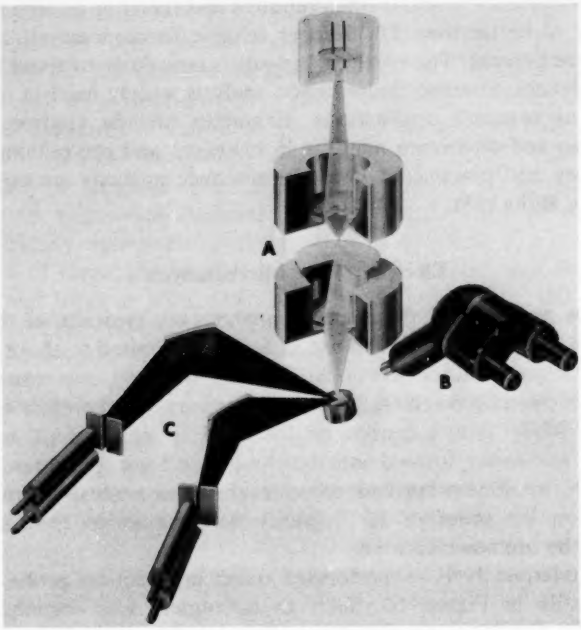

Figure 14 — Schematic of the electron probe microanalyzer (35). The electron optics (a) produce a beam to excite x-rays from a one micron diameter spot on the specimen chosen by use of the light optics (b). Measurements with the x-ray optics (c) yield the quantitative chemical analysis.

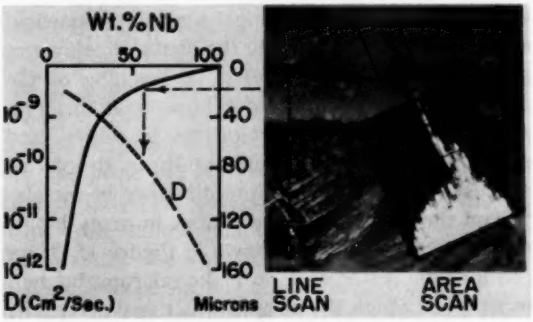

Figure 15 — Titanium-niobium interdiffusion couple measured with the electron probe x-ray microanalyzer. Left: Measured concentration profile and calculated interdiffusion coefficient. Right: Photomicrograph of the diffusion zone plus a composition map of Ti in a grain boundary diffusion feature.

The level of accuracy presently attainable with the electron probe (1-3 percent relative) is set by the complexity of electron-solid interactions. It is difficult to properly consider all important physical aspects of x-ray generation by electrons, even for those processes which are understood. Much of the basic cross section data is not available.

The most physical and general treatment of electron impact x-ray generation is the Transport Equation Program (TEP) developed by Dr. D. B. Brown, now at NRL (36). This program provides a versatile method to calculate the motion of electrons through a solid and the resultant characteristic x-ray production. Presently the program includes consideration of the major interactions determining electron scattering and energy loss. As the less important processes are understood and needed data become available, the program can be expanded quite readily. The TEP in the microprobe field is analogous to the Fundamental Parameters method for fluorescence x-ray analysis. Since most x-ray generators in the laboratory involve electron impact, as in x-ray tubes, the TEP is applicable to calculation of a broad range of x-ray spectra. Currently, Brown is using the TEP to calculate bremssthralung continua, making it possible to obtain the theoretical equivalent of the measured spectrum in Figure 3.

While basic problems concerning electron probe x-ray microanalysis remain, the technique is already widely used. In fact, its popularity in the fields of mineralogy and biology as well as metallurgy approaches that of the electron microscope. It is common for analytical laboratories to have both electron microprobe and fluorescence analysis equipment. Mr. E. J. Brooks uses these instruments in the Central Materials Research Activity at NRL to provide data for a wide variety of research projects.

Ion Excitation of X-Rays

Almost all x-ray spectrochemical analysis has been done with x-ray and electron excitation. Analysis using proton excitation was tested in the mid 1960's at a number of laboratories and seemed to offer little advantage compared to conventional x-ray generation methods (37). Recently, however, it was shown that new ionization mechanisms occur during heavy ion impact, leading to copius x-ray production. Ions of C, O, Ne and Ar from a Van de Graaff accelerator for instance, do not penetrate deeply into solid targets. X-rays are thus generated near the surface. This and the favorable intensity make possible the analysis of surface contaminants, for example, oxidation and corrosion products. Partial monolayers may be measured quantitatively in some cases. Ion excitation surface analysis with x-rays complements the in-depth x-ray analysis which is commonly done with the fluorescence and microprobe techniques. Much fundamental work remains to understand heavy

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ion bombarding effects such as enhanced and multiple ionization, and to determine the depth in a material at which x-rays are generated under ion impact. Basic and associated applied research aimed at evaluating the advantages and limits of ion excitation surface analysis is just beginning using the NRL accelerators.

Bonding Studies Using X-Ray Spectra

We mentioned earlier that valence band soft x-ray spectra are sensitive to the way in which atoms are chemically bound in solids and molecules, as shown in Figure 13. These spectra can thus be used for determination of the arrangement of atoms in a sample. This allows analysis for the compounds present in an unknown, in contrast to the elemental measurements done with hard x-rays in ordinary spectrochemical analysis. But soft x-ray spectra have low intensity and are not best used for analysis of unknowns.

The major value of valence band spectra lies in their use to understand the electronic structure of materials. Figure 16 emphasizes the central importance of electronic structure. The energies and spatial distribution of bonding electrons are important because they determine the practical properties of interest in material applications. Defect as well as perfect crystal properties are ultimately dependent on electronic structure. These include the mechanical, electronic, magnetic, optical, acoustical, thermodynamic, and chemical behavior of components in operational devices and system.

Valence band x-ray spectra have been measured for over 30 years. Much of the early data has been superceded in the past 10 years as capabilities for generating and detecting soft x-rays improved. Both emission and absorption spectra are sensitive to bonding effects. Such

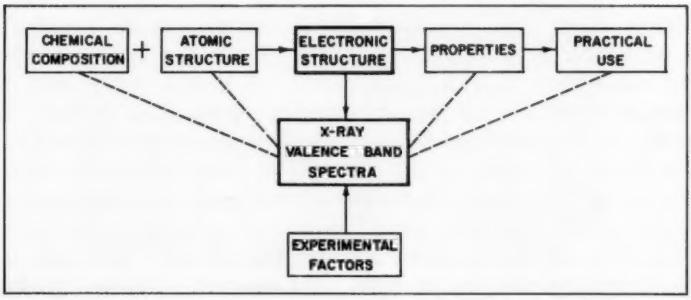

Figure 16 – Relation of material characteristics (top row) to the factors important in the generation and measurement of valence band x-ray spectra (center column). Observed correlations between x-ray spectra and material characteristics are indicated by dashed lines.

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spectra from a wide variety of materials are currently being measured in several laboratories. Since bonding information cannot be derived directly from x-ray spectra it is necessary to use band and bond theories of electronic structure in conjunction with x-ray data. Due to the widespread availability of large, fast computers in recent years, the needed theoretical tools are now available. Combined theoretical and experimental soft x-ray studies of electronic structure are underway at NRL (38).

X-Ray Spectroscopy of High Temperature Sources

Although the study of high temperature plasma sources by means of their x-ray emission began much later than atomic and solid state x-ray studies, x-ray data have already proved to be highly useful. Plasmas, which are ionized gases of energetic electrons and ions, constitute the fourth state of matter. Figure 17, constructed by Dr. A. C. Kolb and Mr. E. A. McLean, shows that plasmas are actually very common in nature, ranging from the coldest, least dense interstellar matter to hot, compact stellar interiors. Man-made plasmas fall between these extremes. Those plasmas with temperatures above a 10^6°K can be studied by means of their x-ray emission.

Whatever the plasma, natural or man-made, three quantities are of interest in the measurement of radiation from hot sources. These are the spatial, spectral and temporal distribution of x-ray emission. They are important because of what they imply about the properties and mechanisms of a source. That is, x-ray data can be used to determine the density, thermal history and opacity of a plasma, and whether the emission is due to bound-to-bound, free-to-bound, thermal bremsstrahlung, synchrotron, or Compton processes. Since x-rays cannot be focussed by lenses in a camera, spatial information on the location of x-ray sources is usually obtained by pinhole or reflection cameras, or by the use of collimators which work like the blinders on a horse. Spectral information (photon energy and intensity) is gotten with instruments such as shown in Figure 6. Time variation is mainly studied with active electronic detectors.

In this section, x-radiation from laboratory plasmas will be briefly discussed. The measurement of hot sources which occur naturally in space, that is, x-ray astronomy, comprises a large field which must be treated separately. Laboratory and astronomical measurements of plasmas are complementary. Earth-bound experiments can be controlled and yield information useful for interpreting astrophysical data, for example, spectra line energies. On the other hand, the continuous cosmic sources provide conditions which cannot be duplicated in the laboratory.

There has been a great deal of basic work in the past 15 years aimed at understanding the occurrence, characteristics, and dynamics of laboratory plasmas. The study of plasmas is related to many practical

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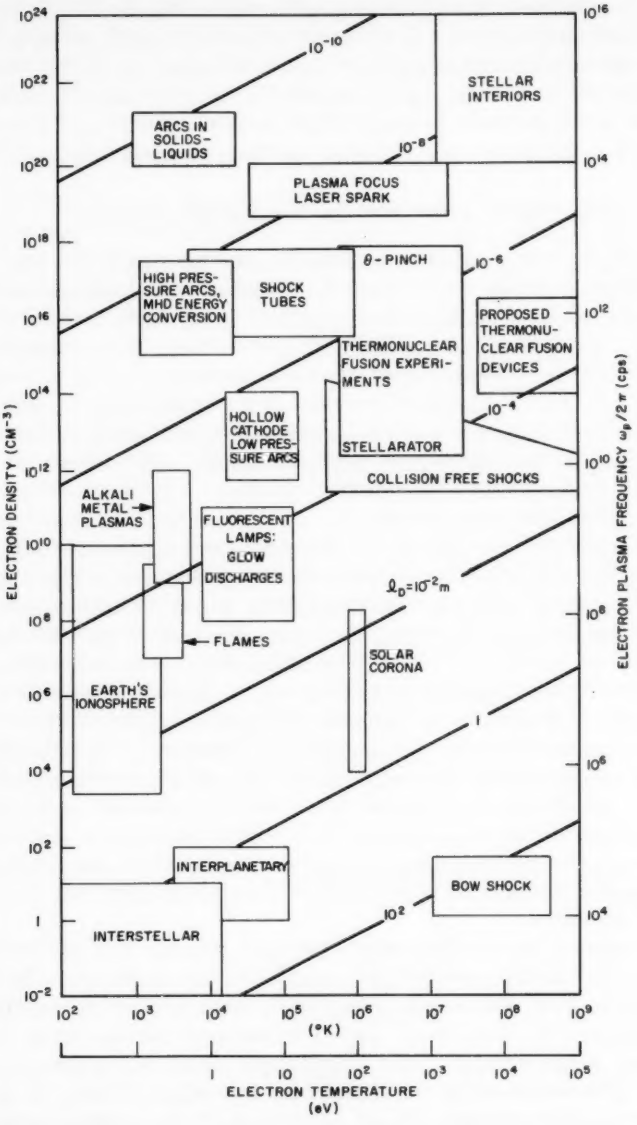

Figure 17 — Electron density and temperature diagram for natural and laboratory plasmas. The plasma frequency and the Coulomb potential screening distance λ_D are also given.

problems in addition to their use for interpreting astrophysical data. These include magnetohydrodynamic power generation, materials processing such as plasma anodization, reentry heating, and chemical kinetics in explosions and shock waves. The field has been especially influenced by the over-riding practical goal of controlled fusion for thermonuclear power generation. Past experience has shown that the Navy makes use of all major power sources, for example,nuclear fission. Similar use of fusion power can be expected if it becomes feasible. Work on and related to the fusion power problem has been pursued at NRL for about ten years mainly under the direction of Dr. A. C. Kolb.

In order to generate power by fusion, it is necessary to confine a high density plasma at high temperatures for relatively long times. Instabilities limit plasma containment times. To understand the behavior of a plasma and its instabilities, it is necessary to know the electron density and temperature as a function of time. These parameters, especially temperature, determine the ionization state of plasma constituents. A large number of diagnostic techniques are used to determine plasma behavior. These are reviewed in a book by Dr. A. R. Griem, formerly at NRL (39). Time-dependent UV and x-ray spectroscopy of emission from various ion species in a plasma allows determination of the temperature history. An example is given in Figure 18 (40). X-ray emission occurs primarily near the time of peak heating and is

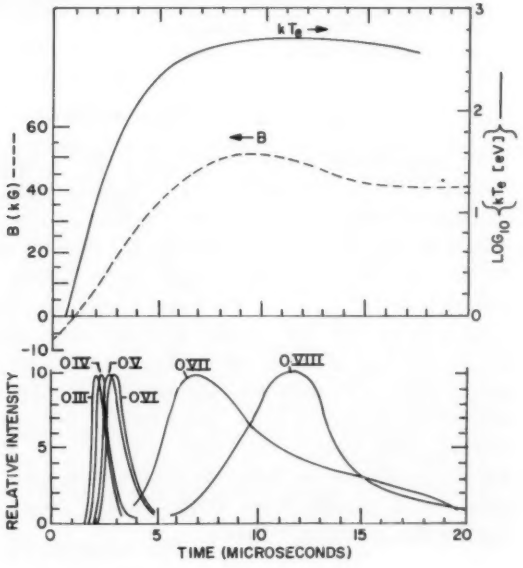

Figure 18 —Time dependence of oxygen impurity emission lines (bottom) and of unabsorbed x-ray continuum intensity (not shown) give the electron temperature history (top) in a high energy plasma. The confining magnetic field is included for comparison (40).

therefore useful in calculating the extreme conditions attained in a plasma. Lie and Elton recently observed that a spark discharge produced H-like Ti (41). This and lines from other ionization states are shown in Figure 19. The data indicate a temperature of 10^{7}°K and a density of 10^{20} electrons/cm^{3}.

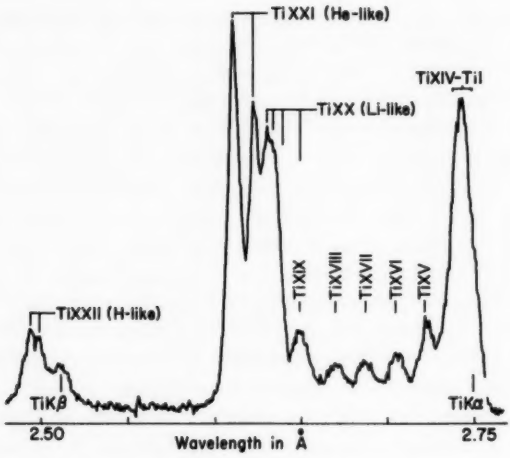

Figure 19 — Densitometer tracing of the time integrated x-ray spectrum from the plasma formed during a vacuum discharge with a Ti anode. Radiation is observed from several highly ionized states, including Ti stripped of 21 of its 22 electrons (41).

X-Ray Astronomy

In earth-bound x-ray experiments the radiation is generated and absorbed in times of the order of 1-10 nanoseconds. Compared to this, x-ray astronomy might be called the "science of old x-rays." The sources of x-rays in space range from the sun, 8.3 minutes away at the speed of light, to stars on the order of 10^{3} light years distant, to galaxies from which x-rays take 10^{8} years to reach our planet. In contrast to the age of some x-rays measured, the science of x-ray astronomy is only 21 years old. Its advent had to await the availability of rockets which could lift instruments above the absorbing atmosphere. Rocket spectroscopy in the UV region was begun by NRL scientists in 1946 using captured V2 rockets. X-rays from space were first measured in 1949 NRL experiments. Figure 20 shows an Aerobee rocket now used for x-ray astronomy in shots fired usually from White Sands, New Mexico and less often from Wallops Island, Virginia.

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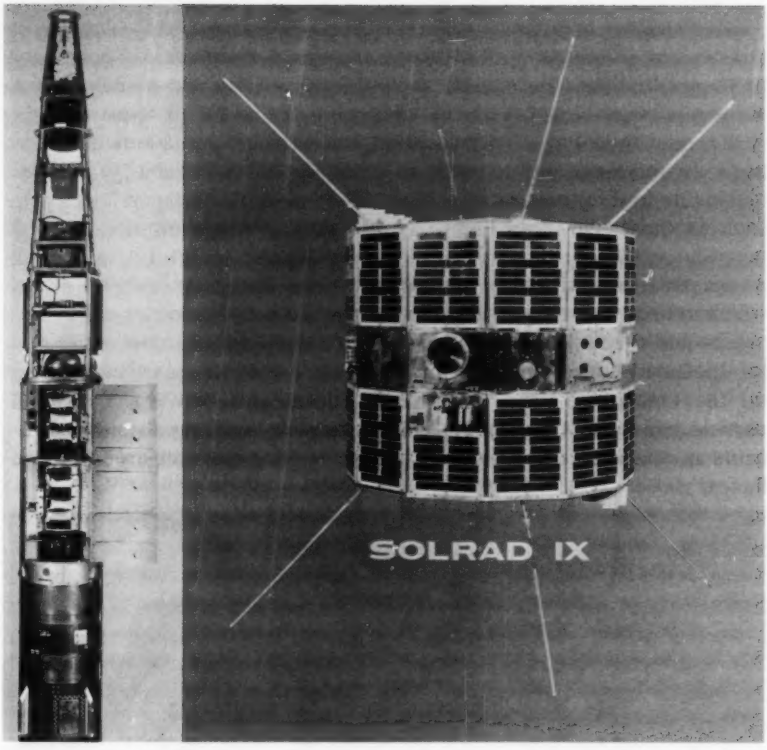

Figure 20 — Aerobee rocket payload (15 in. diameter) and SolRad 9 satellite (30 in. diameter) used for measurement of cosmic x-ray sources

Rocket flights have served two purposes, yielding important results as well as providing tests of eqiupment later used on satellites. Since the observation time from rockets is short, about five minutes per flight, satellites have provided a large increase in observational capabilities. NRL has had its own Solar Radiation (SolRad) satellites in orbit since 1960. The SolRad 9 satellite, which was launched 5 March 1968 is shown in Figure 22. Many NRL experiments have also been flown on NASA satellites such as the OSO's, Orbiting Solar Observatories.

Some recent examples of spatial, spectral and temporal x-ray astronomy measurements of the sun and more distant sources will be given in the rest of this article. The information obtained contributes to problems as diverse as reliable radio communications and the evolution of the universe. This work has been done and continues in the Space Science Division and Hulburt Center for Space Research under the direction of Dr. Herbert Friedman, with support from the Space Technology and Engineering Services Divisions at NRL.

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Solar X-Ray Astronomy

The sun is, of course, a critically important astronomical object for us on earth. Most solar energy is emitted in and near the visible region, although the solar spectrum extends from the x-ray to radio regions. The visible radiation is very constant, while great fluctuations in output occur in the x-ray and extreme UV regions. This variability provides a valuable tool for understanding processes in the outer layers of the sun such as those shown in Figure 21. Solar x-ray emission also has tremendous effect on the earth's ionosphere. Together with UV radiation, x-rays are responsible for causing variations in the ionospheric layers which reflect and attenuate radio waves. Understanding of solar processes and the ability to predict x-ray outbursts are therefore important for the operation of long range radio systems which bounce radiation off the ionosphere. Also because of correlations between x-ray and particle emission, x-ray measurements provide a means to alert astronauts in space of the coming of slower-moving but dangerous proton fluxes.

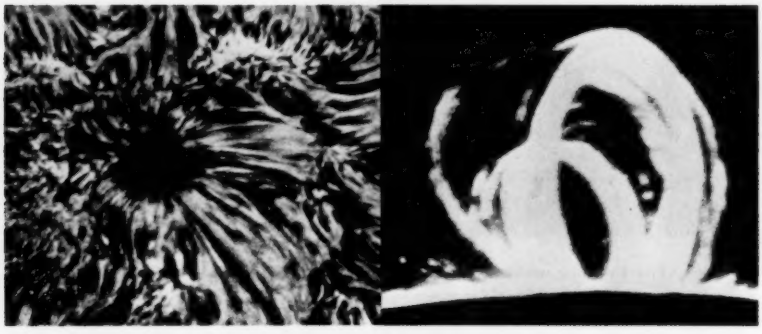

Figure 21 — Sunspot (left) and limb (right) photographs of the sun showing processes associated with x-ray emission

Solar x-ray spectroscopy has been pursued at NRL in a wide variety of rocket and satellite experiments. The rocket work has resulted in many "firsts," both in the instrumentation used and the results obtained (42). Solar x-ray emission and its role in production of the ionosphere were discovered in 1949. Measurement of x-rays during a solar flare in 1956 showed that x-rays, not UV radiation, cause radio fadeout. A rocket flight during an eclipse two years later proved that x-rays arise from local, active regions. The initial x-ray image of the sun, obtained in 1960, showed that areas of x-ray activity are also associated with regions of exceptional activity in the visible and microwave regions (43). This pinhole camera image and a more recent example obtained at longer wavelengths (44) are shown in Figure 22. The earliest high

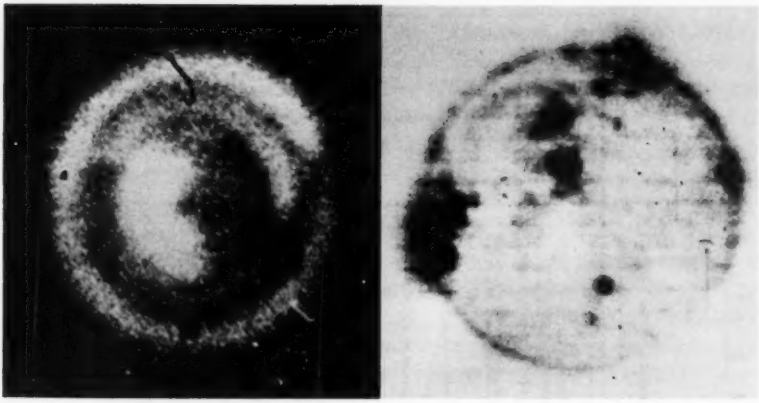

Figure 22 — The first solar x-ray image (left) taken with a pinhole camera, was blurred by rotation of the rocket (43). A more recent soft x-ray image (right) was obtained by reflection from a mirror covered by an Al filter (44). Areas emitting x-rays appear white on the left image and black on the right image.

resolution solar x-ray spectra were measured using a crystal spectrometer on an Aerobee rocket flight in 1963 (28).

Spectral and especially time variation studies have been done best using satellites. A high resolution spectral scan (45) from the OSO-6 satellite is given in Figure 23. Lines from several elements in high states of ionization are evident. An energy sensitive detector on the same satellite yielded the flare data (45) in Figure 24. The more energetic radiation tends to peak earlier and decay more rapidly than the softer component.

The NRL SolRad satellite program, under the scientific direction of Mr. R. W. Kreplin, is also producing highly time-resolved data (46). Spectral sensitivity in SolRad x-ray experiments is obtained by using detectors with various combinations of windows and filling gases, giving them different pass bands. The data in 3 bands for the first few months after launch of SolRad 9 is shown in Figure 25. The SolRad program has been especially valuable because it has spanned over one-half of the 11 year solar activity cycle. The x-ray intensity varied in the 8-20 Å band, for example, by a factor of 200 between the 1964 activity minimum and the 1969 maximum (47). The SolRad vehicles are built at NRL by the Satellite Techniques Branch under the direction of Mr. P. G. Wilhelm.

While the SolRad data is very valuable for basic studies of the sun, its most pressing use is for prediction of disturbances which can affect communications, astronaut activity and military operations. The aim

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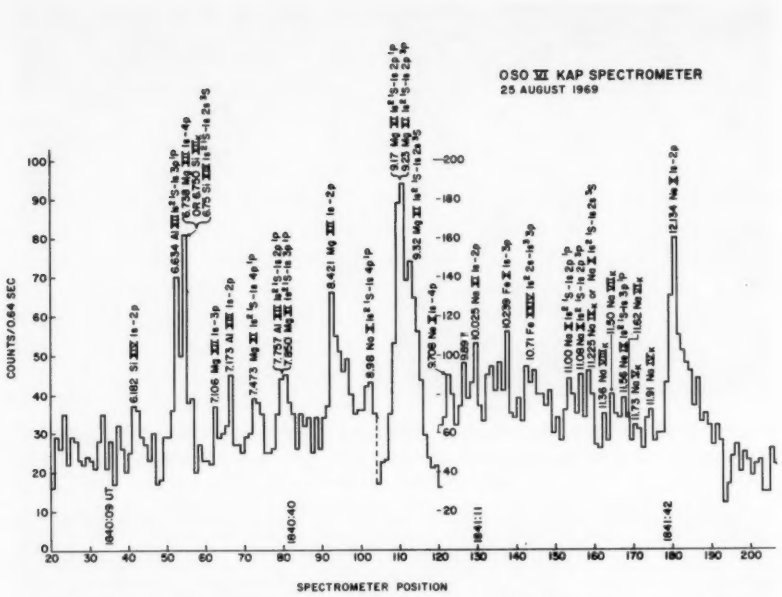

Figure 23 — High resolution solar x-ray spectrum obtained with a KAP crystal spectrometer aboard the OSO-6 spacecraft. Wavelengths (Å) shown are obtained from spectrometer position calibration. Time during the spectral scan is also shown (45).

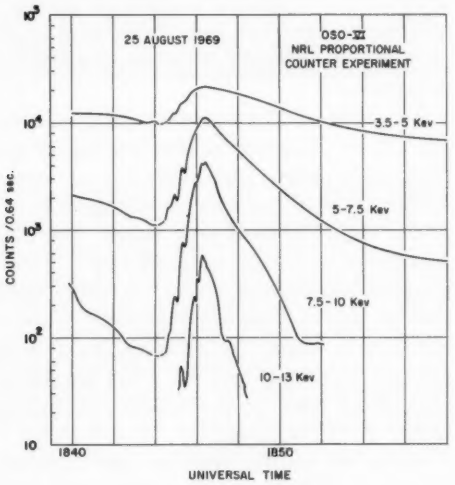

Figure 24 — Solar flare activity measured from the OSO-6 satellite using a proportional x-ray counter (45)

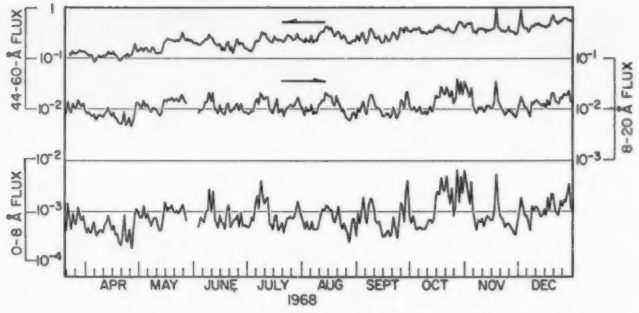

Figure 25 — Intensity variation in three x-ray bands measured during the first months of the SolRad 9 satellite operation. Intensity is given in ergs/cm²/sec (47).

is to use current x-ray intensity information to anticipate both x-ray flares which affect the ionosphere, and high-energy proton fluxes which might endanger personnel in space. Early SolRad data were intermittent, but now continuous data are available. The increased amount of information and especially its use for radio blackout and proton flux predictions necessitates rapid data analysis and dissemination. A system for this purpose has been designed and is being operated and refined by NRL personnel with Mr. E. W. Peterkin as Technical Project Manger. The Operational Research Branch under Mr. C. H. Chrisman is responsible for data handling. In addition to promulgating routine flare warnings, the SolRad team has already provided x-ray intensity watches during four Apollo lunar flights. Figure 26 shows the relation of all elements in the SolRad system.

The solar x-ray studies highlighted in this section have contributed greatly to the current understanding of solar emission processes (48). Also, there is on hand a large amount of information against which to test developing theories. Many fundamental questions remain, even including the reason for the 11-year length of the solar cycle. While our understanding of the sun develops, x-ray data will be used increasingly for practical solar and ionospheric weather forecasting.

Night Sky X-Ray Astronomy

Measurement of x-rays from distant sources in the night sky began in 1962 (49). Activity in this field is intense and competitive. The work at NRL by and under Dr. Friedman has gained wide respect for its breadth and quality. The problems in this area are similar to those in solar studies, determination of the location, spectra and time variation of x-ray sources. These sources include stars, galaxies and possibly hot intergalactic gas. Results obtained have contributed to knowledge

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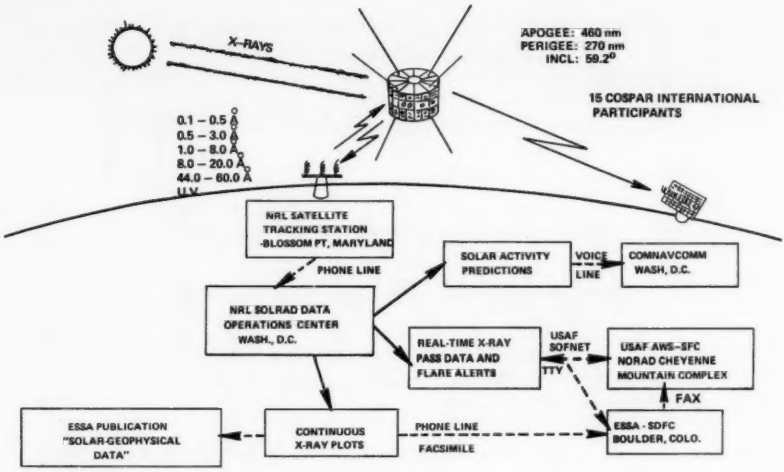

Figure 26 – The SolRad 9 solar x-ray monitoring and disturbance warning system

of stellar and galactic evolution as well as the development of the entire universe.

At present about 35 localized sources of cosmic x-rays are known. The intensities of these distant sources are small compared to those from the sun. Figure 27 shows the distribution of sources in galactic coordinates. Most x-ray stars lie near the galactic plane, indicating that they are part of our galaxy, the Milky Way. Almost all of the sources shown in Figure 27 were initially located by NRL rocket surveys performed by Friedman and colleagues (50). In addition to locating many x-ray sources, this and subsequent work showed several of them to be highly variable. Only a few of the x-ray source positions are known well enough for certain association with an optical object.

An x-ray source in the Constellation Taurus, Tau XR-1, is one of the strongest and most studied. It has been identified with the Crab nebula, the material thrown off by a star which exploded so brightly in 1054 A.D. that it was observable even in the daytime by oriental astronomers. X-ray emission from this nebula was discovered in 1963 by Bowyer, Byram, Chubb and Friedman (51). About one year later x-ray detectors were flown on a rocket simultaneous with a lunar occultation of the nebula in order to determine the size of the x-ray source (52). Figure 28 shows the nebula in relation to the limb of the moon during the flight. X-rays from the source were extinguished slowly, indicating that x-ray emission originates in a broad region. This ob-

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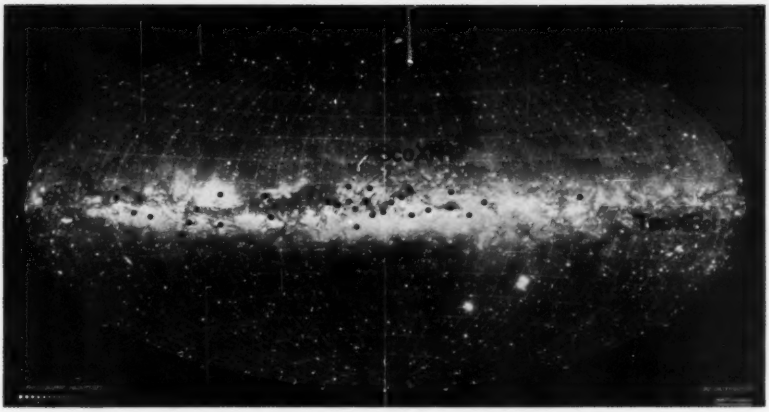

Figure 27 — The location of night sky cosmic x-ray sources plotted in galactic coordinates over a composite all-sky photograph made at the Lund Observatory. The three sources discussed in the text are labelled (49).

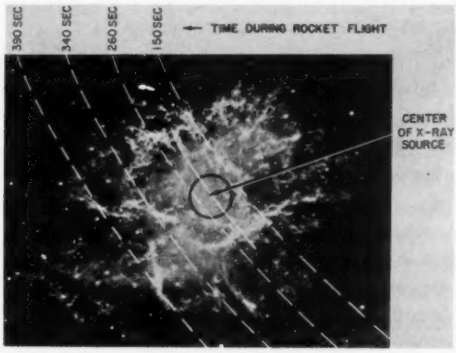

Figure 28 — The Crab nebula and lunar position during the occultation experiment which proved that the x-ray source in the nebula is broadly extended, as shown (52)

servation seemed to rule out a point source of energy within the Crab nebula.

It was discovered in 1968 that some of the radio and optical radiation from the nebula pulses at the rate of 30 times a second. Optical intensity as a function of time is shown in the upper part of Figure 29. Pulsations in the x-ray region were first found by an NRL team on a flight only two months after the optical pulsar was reported (52). The data obtained by Fritz, Henry, Meekins, Chubb and Friedman is also shown in Figure 29. The pulsed x-ray component of the Crab amounts to about 5×10^{35} ergs/sec, compared to the 4×10^{33} ergs/sec total steady energy output of the sun.

Association of a pulsar with the Crab nebula has reopened the question of a point energy source. It is now hypothesized that a neutron star

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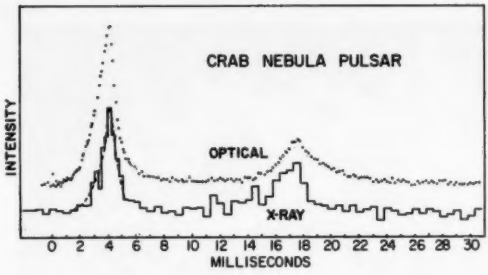

Figure 29 – Time variation of optical and x-ray pulsar emission from the Crab nebula (53)

consisting of about one solar mass in a sphere only 5 miles in diameter, the collapsed remains of the stellar explosion, exists in the nebula. The rapidly rotating and nonuniform neutron star throws off gas at relativistic velocities. The interaction of this material with magnetic fields in the nebula produces x-rays over a broad region, consistent with the occultation measurement. The postulated synchrotron radiation mechanism is also consistent with the observed spectrum.

While the Crab nebula is a fascinating and fruitful object for study, several of the x-ray sources have also yielded valuable information. Only two more can be mentioned here. The brightest x-ray source is in Scorpio at the distance of about 800 light years (54). This object, Sco XR-1, emits 99 percent of its energy in the x-ray region so that the optical object associated with it is very faint. Spectral measurements of radiation from Sco XR-1 are consistent with a thermal bremsstrahlung source at a temperature of about 6×10^{7}°K. The x-ray and optical emission vary irregularly. Binary star models in which gravational energy is released as the more dense partner acquires matter from the other have been proposed as the energy source for Sco XR-1.

X-ray galaxies, as well as x-ray stars, have been discovered by NRL astronomers. The most intense of these is M-87 in Virgo, 10^{8} light years distant (54). This galaxy emits 70 times more x-ray energy than energy in the radio region. Since the intensity from M-87 is low, its spectrum has not been established. Locating and characterizing x-ray galaxies continues as a major goal of x-ray astronomy work at NRL.

In general, the number of x-ray counts which can be accumulated limits both the discovery of low intensity discrete sources and spectral determination of known sources. Given the limited payload and observation time in a rocket flight, greater counter sensitivity is indicated. No quantum increases in sensitivity are possible, however. Satellites with their long observation times will provide the solution to count accumulation problems in night sky x-ray astronomy. The better spatial resolution which can be attained using fine collimators, if long counting times are possible, would be especially valuable for study of the diffuse x-ray background from space.

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Cosmic x-ray background radiation in and perpendicular to the galactic plane was recently measured by Henry and collaborators (55). Radiation from the pole can be split into two contributions. The first is an extrapolation of the polar radiation observed at higher energies which eventually curves over due to absorption by interstellar matter within the galaxy. The second, an excess of low energy radiation, has been attributed to bremsstrahlung from a hot intergalactic gas with a density 10^{-5} hydrogen atoms/cm^3 and a temperature of about 4×10^6°K (56). This interpretation has an important cosmological consequence. It implies that there is enough matter in the universe to provide a gravitational deceleration sufficient to halt its outward expansion. The elapsed time from the "Big Bang" postulated to have occurred about 10 billion years ago until the cessation of expansion is calculated to be about 40 billion years. For comparison, the earth is roughly 4.5 billion years old.

Another fascinating result of the soft x-ray observation concerns the distribution of matter in the universe. The average mass density of all galaxies is estimated at about 1/30th of 10^{-5} H atoms/cm^3, that is, only about 3 percent of the matter in the universe is in optically observable galaxies. If this is true, almost all of the matter in the universe is detectable only because of its x-ray emission.

It is interesting to note that the alteration at low energies of the spectrum from a known cosmic source at a known distance allows estimation of the nature and density of interstellar matter. This is, in essence, an x-ray chemical analysis of a tenuous but immense cosmic "sample." It is also a form of radiography, which brings us back to the first x-ray application discussed in this article.

Conclusion

It has been possible to treat only highlights in the several areas of Navy x-ray research and application. It has not been possible to adequately cover the work in neighboring spectral regions which shares techniques and goals with x-ray studies and applications. Reference is made to work by Dr. R. Tousey in the UV region (57) and gamma ray work in the Nuclear Physics Division at NRL (58).

We have reviewed the basic physical qualities of x-rays. Each has been exploited in a major area of x-ray work. The applicable equations and associated fields are summarized in Table 2. X-ray research in all of these areas has a rich history. In particular, most of our knowledge of atomic and electronic structure derives from x-ray diffraction and spectroscopy experiments. It seems clear that modern technology could not have developed as rapidly as it did without this knowledge.

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TABLE 2
Areas of X-Ray Application Associated with Physical Characteristics of X-Rays
E = x-ray photon energy
E(kev) = 12.348/λ (Angstroms)

Equation	X-Ray Characteristic	X-Ray Field
Absorption Coefficient $\sim C/E^3$	C relatively small for hard x-rays. X-rays are penetrating.	Radiography
Number of Ion Pairs $\sim E/\epsilon \sim 10^3$	E large compared to ionization energy ϵ. X-rays absorption produces defects.	Radiation Damage and Dosimetry
$\lambda = 2d \sin \theta$ d = interplanar spacing θ = Bragg angle	$\lambda \sim$ atomic size and spacing. X-rays scatter coherently.	Structure Determination
$E = E_2 - E_1$	E_2 and E_1 = atomic energy levels. X-rays are characteristic of the emitting atom.	Spectroscopy of cold sources
$E = kT$ k = Boltzman's Constant	$T \sim 10^7$°K. Very hot sources emit x-rays.	Spectroscopy of hot sources

Present x-ray research consists largely of establishing the applicability and capability of x-ray methods for the study of materials. How large a molecular structure can be determined, and to what accuracy, using x-ray diffraction? What aspects of bonding electron energies can be extracted from valence band x-ray spectra? What is the utility of x-ray interferometric techniques which are capable of detecting displacements on the order of the size of atoms (10^{-8} cm)? These and similar large questions are currently being addressed.

Also important is the comparison of x-ray methods with other techniques. Discussed in this article are x-ray techniques which yield information on the chemical composition, perfect and defect atomic structure, and electronic structure in a very broad range of materials. This arsenal of methods is heavily used for the characterization of new materials. However, x-ray measurements are certainly not optimum for all work where they can be used. It is necessary to know which experimental approach best solves a specific materials problem, as well as the limitations of the method. To cite some examples: (a) numerous methods exist, other than x-ray spectrochemical analysis, for the determination of chemical composition, (b) neutron and electron dif-

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fraction are more useful than x-ray diffraction for some structure studies, and (c) several complementary and competing experimental methods for determining electronic structure are under development and in use. Comparison of x-ray and other methods is progressing as our knowledge of each of these tools develops.

Other current x-ray work is concerned with fundamental questions in the more recently developed areas. X-ray astronomy, especially, is rife with basic questions. Much definitive work also remains concerning x-ray processes associated with high energy particles. The basic mechanisms of x-ray excitation with MeV energy ions are not entirely understood. The transition x-radiation emitted when superhigh energy (GeV) particles enter a metal from a vacuum has only recently been observed. Figure 30 shows an example from the work of Yuan at Brookhaven (59). Such spectra are potentially useful for measuring high particle energies. In fact, x-ray emission due to several mechanisms attends the operation of high energy accelerators and the radiation from many machines has been little studied and exploited.

Beyond the established techniques undergoing detailed examination and the newer areas whose characteristics are being defined, there will probably be other major x-ray problems requiring future attention. One of these could be an x-ray laser (xrayser). Whatever the eventual progress toward such a device, the point of diminishing returns for x-ray research is not in sight.

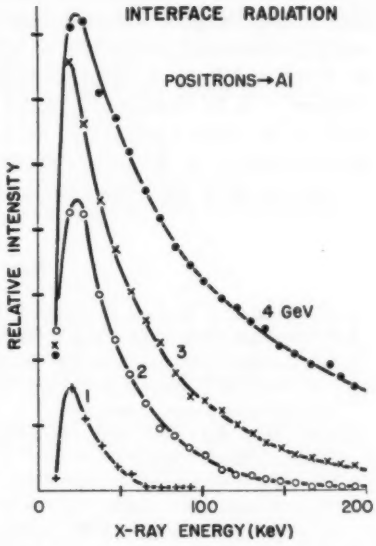

Figure 30 — X-ray spectra emitted by high energy positrons while moving from a vacuum into Al metal (59)

While progress is being made toward answers to present x-ray research questions, there is an accelerating practical payoff because use of x-ray methods is rapidly increasing in many fields. Work in several of them has been mentioned in this article, for example, physics, chemistry, astronomy, biology, medicine, and materials engineering. Many industrial as well as military applications have resulted from Navy x-ray research. An example is the use of x-ray intensity to monitor tin plate thickness in sheet mills. Others have been mentioned. These benefit the Navy as a user of materials, as well as the country as a whole.

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The Navy necessarily has a strong interest and capability in developing techniques and using radiation in all regions of the electromagnetic spectrum. The nature of this interest varies. Only three regions, optical, radar, and radio, are heavily used for the operation of submarines, ships, airplanes and satellites. Work in the other spectral areas mainly plays a critical supporting role, contributing to the materials and power sources necessary to operate the fleet. The precise short and long term value of answers obtained from x-ray research and applications may be difficult to judge in advance. However, the scientific, industrial, and military value of past Navy work with x-rays is indeed evident and there are strong reasons for believing that the current work in the x-ray region will also prove to be most useful. The promise is especially great for the increased use of x-ray methods in the development and rapid testing of the new materials needed to improve the Navy's operational capabilities.

Acknowledgements

Instruction and inspiration from L. S. Birks and J. Karle are gratefully acknowledged. Valuable help was also received from others whose work is discussed, especially F. H. Attix, E. L. Criscuolo, R. C. Elton, G. Fritz, and R. W. Kreplin. Illustrations were provided by: J. V. Gilfrich (Figure 3), J. D. Bultman (7), R. H. Fuller (8), F. H. Attix (9), I. L. Karle (10), L. S. Birks (14), E. A. McLean (17), R. C. Elton (18,19), E. T. Byram (20), P. G. Wilhelm (20), H. Friedman (21,22,28), R. W. Kreplin (23,24,25), E. W. Peterkin (26), and G. Fritz (27,29).

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Gun Electron Injection for Semiconductor Hybrid Amplification (GEISHA)

Max N. Yoder
Office of Naval Research

The Office of Naval Research (ONR) is currently engaged in a research and development task known within the Department of Defense as CHAIR GEISHA. By way of definition, CHAIR indicates that the task was conceived by and is being administered by ONR. GEISHA is an acronym for the descriptive title above. This venture is expected to vastly increase the mean-time-to-failure for both military and commercial electromagnetic systems and to provide electronic engineers with an electronic building block exhibiting greater versatility and flexibility than previous means. This article is intended to acquaint the reader with the basic fundamentals of this new technology.

Basic Fundamentals

By definition, a semiconductor is a material which is a much poorer conductor of electricity than is, for example, a copper wire. In contrast, however, a semiconductor is a much better conductor than an insulator such as glass. In its most common use, a semiconductor is a crystal of silicon or germanium—both of which are elements. Much can be done to modify the electrical properties of a pure crystalline semiconductor. To enable the semiconductor to be a better conductor of electricity, various impurities may be "mixed" with it. This process is called "doping." Two approaches are commonly used. The first introduces dopants which cause the semiconductor to have excess electrons within its atomic structure. As such, it may be thought of as having excess electrons within its atomic structure. As such, it may be thought of as having excess negative charge. Semiconductors doped in this manner are known as n type semiconductors. A second approach uses impurity dopants which introduce vacancies or holes in the structure of the crystal—holes that would have been filled with electrons if the semiconductor had not been doped. These holes or absence of electrons may be thought of as places of positive charge. Hence, semiconductors doped in this manner are known as p type semiconductors. Semiconductors can be doped before or after the crystals are grown. The most commonly used method of doping after growth is known as diffusion. In this method, the impurity is placed on top of the semiconductor crystal and the combination is placed in an oven. The temperature and

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the duration determines the depth and concentration to which the impurity diffuses into the crystal.

Should a crystal be grown first with n type impurities and then diffusion doped with p type impurities, there will exist within the crystal a region near the surface which is p type while the remainder is n type. By definition this is a p−n junction and exhibits the properties of a vacuum tube rectifier. When a semiconductor p−n junction is reverse biased by battery E_b, no electrons flow through the circuit shown in Figure 1. The current (I) is zero, and from Ohm's Law, no voltage is developed across load resistor R_L.

A new method of doping semiconductors was recently developed such that the p region could be made very thin (*e.g.* $< .5 \times 10^{-4}$ cm). Thus the p−n junction can be very shallow as shown in Figure 2. (The advantages of this will be apparent later.)

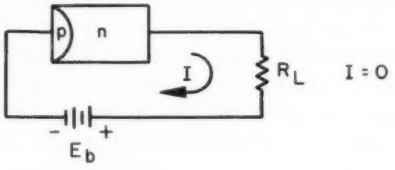

Figure 1 — Reversed biased diode

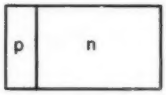

Figure 2 — Ion implanted junction

The ion implantation technique consists of accelerating positive ions of boron or aluminum to very high velocities and letting them impact into the n type semiconductor. The penetration depth is directly proportional to impact velocity and thus the junction depth can be more precisely controlled than by the common thermal diffusion method of semiconductor manufacture. A heavy concentration of boron or aluminum ions results in a p^+ region rather than a p region.

When such a junction is reverse biased, an electric field exists through a portion of the n region which is depleted (by virtue of the bias voltage) of charge carriers (electrons). By applying a backing layer of heavily doped n type material and choosing the geometry and bias voltage correctly, the entire n region can be depleted of electrons and an electronic field (E) made to exist throughout the n region as shown in Figure 3. The addition of a heavily doped n region (noted as n^+) more exactly defines the depletion region and aids in the device operation to be explained.

Should an electron with high velocity strike the reverse biased diode it (being so much smaller and lighter than an ion) will penetrate deeply

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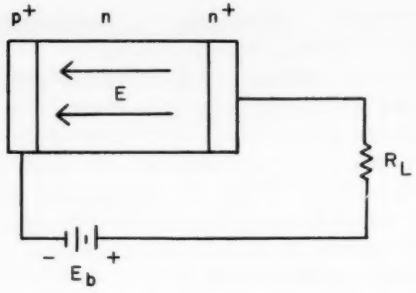

Figure 3 – Depleted diode

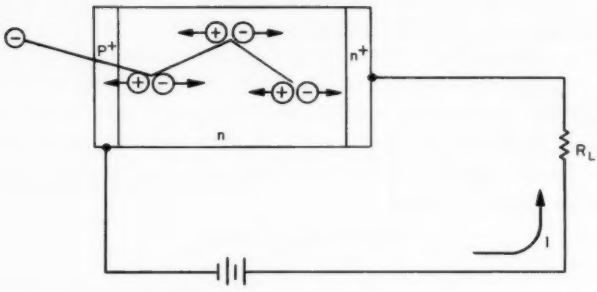

Figure 4 – Electron-hole pair creation

into the semiconductor. In the process of expending its kinetic energy and coming to rest within the semiconductor, the electron will knock many other electrons out of their orbits around their respective atomic nuclei. This process is referred to as impact ionization of the lattice structure. Atoms with electrons missing are ready acceptors of electrons. These electron vacancies (as previously explained) act as "holes" in semiconductor theory. To summarize this action it can be said that an energetic impacting electron creates many electrons and holes (shown as − and + respectively in Figure 4) in coming to rest within the semiconductor. This is referred to as electron-hole pair creation. Since these electrons and holes are created in the presence of the strong electric field E in the depletion region, the electrons are accelerated by the electric field to the positive terminal (n^+) and the holes are accelerated toward the negative terminal (p^+) before they can recombine with each other. Figure 4 illustrates this action.

As holes arrive at the p region of the semiconductor they combine with electrons supplied from the battery. Electrons flowing from the battery to combine with the holes are replaced at the positive terminal of the battery to combine with the holes are replaced at the positive

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terminal of the battery by electrons collected in the n^+ region of the semiconductor. This action of electrons causes a current I to flow in the external circuit. The advantages to be gained by this arrangement are many, but the primary advantage is that a single electron bombarding the semiconductor can cause many thousands of electrons to flow in the external circuit. In fact, for every 3.6 volts (of the battery E_B) that accelerates the bombarding electron, one electron-hole pair is created in the semiconductor. Thus a single electron accelerated through a potential of 15,000 volts prior to impacting the semiconductor will create over 4000 electron-hole pairs in the semiconductor. In turn, over 4000 electrons will flow in the external circuit. Thus this simple device has a current gain of 4000 or in electronic terms, 36 decibels. It can be appreciated that a very small current striking the semiconductor can then be made to cause a very large current in the external circuit. The physical arrangement for so doing is shown in Figure 5. Here electrons are "boiled" off the cathode and accelerated toward the semiconductor by battery E_B. In essence, this is a gun that shoots electrons. The potential voltage E_g on the anode determines the number of electrons per second striking the semiconductor. Thus the E_g voltage determines the electron beam current which in turn controls the output current I. The basic concepts shown above were first disclosed by Moore, *et. al.* (1), and later by Brown (2), and Norris and Gibbons (3). Recent advances in semiconductor processing technology have opened up an even greater potential for using these devices.

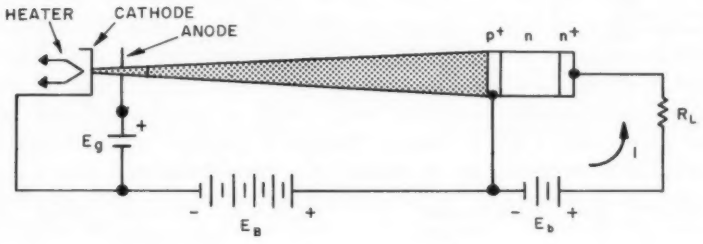

Figure 5 — Simple GEISHA

Vacuum Tube Comparisons

A measure of the quality of an electron tube is made by the determination of the change in the output current as a function of the change of the input voltage. This measure is called the transconductance. In a good ultra high frequency (uhf) triode it is typically between 15 and 20 microohms. In the GEISHA it can be as high as 5000 microohms.

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One of the biggest disadvantages of a conventional high power microwave amplifier tube is that it requires a very high electron beam current (*i.e.* several amperes). This creates two major problems. The first is that the tube cathode is stressed to the extent that its life is shortened. Thus a built-in wear-out mechanism exists. In comparison, the cathode loading of the GEISHA is many thousand times lower—thus substantially increasing its life. A second disadvantage in the high electron beam current of conventional power tubes is that the electrons are so closely packed that the electrostatic forces of repulsion between individual electrons tend to disperse the beam. A bulky magnet is often required to confine the beam. A beam requiring a magnet (with its extra weight and space requirements) is usually referred to as a high perveance beam. In contrast, the GEISHA requires a low perveance beam and no magnetic field. Thus a smaller, lighter, simpler device is possible.

Transistor Comparisons

If the polarity of the battery E_b of Figure 1 is reversed, then electrons will flow continuously in the external circuit—even in the absence of external electron bombardment ionization. By definition, a p—n junction so biased is referred to as being forward biased. Such a circuit is shown in Figure 6. An equivalent circuit is shown in Figure 7.

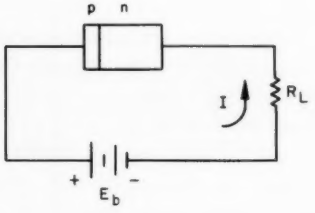

Figure 6 — Forward biased diode

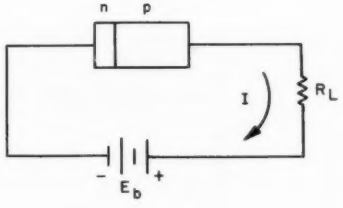

Figure 7 — Equivalent, forward biased diode

A transistor is created by placing two p—n junctions back to back as shown in Figure 8. Actually, there is but one p region which is common to both junctions as shown in Figure 9. Figure 10 shows the first n—p junction being forward biased. Electrons depart the negative terminal of battery E_c, pass through the input resistor R_{in} and into the n region of the semiconductor. They continue through the n—p junction and into the p region and back to the positive terminal of the battery. If the p region is made very thin, many of the electrons injected into it from the

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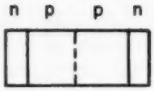
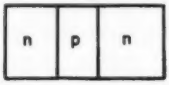


Figure 8 — Transistor formation

Figure 9 — An n–p–n transistor



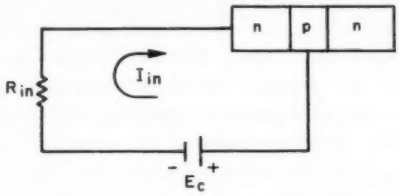


Figure 10 — Forward biased emitter-base junction

n region pass through and into the n region on the opposite side where they are collected. Figure 11 is illustrative of the process. Here it is seen that the n–p junction is reverse biased by battery E_b in exactly the same manner shown earlier in Figure 1. As such, there is no output current (I_{out}) unless electrons are injected into the depletion region in the n portion of the semiconductor as was explained earlier. The forward biased n–p junction supplies the required electrons as was explained above. Thus a transistor operates by the n region emitting electrons into the thin p region with most of them diffusing through the p region and into the opposite n region where they are collected. The n region which emits the electrons is logically known as the emitter while the n region collecting the electrons is known as the collector. The center p region has been tagged the base. Inasmuch as the battery E_b is normally of higher voltage than that of battery E_c and since (in the circuit shown)

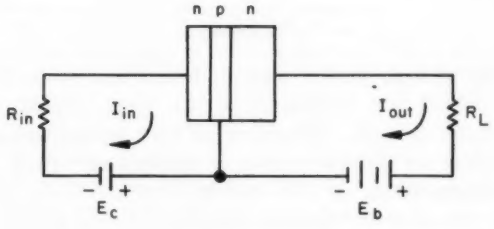


Figure 11 — Transistor circuit

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the input current (I_{in}) and the output current (I_{out}) are of approximately the same amplitude, the output power is greater than the input power. Thus it is an amplifier—and a very compact one at that. In fact, it is considerably smaller than the GEISHA. The GEISHA is, in reality a transistor with the emitter removed and replaced by an electron gun.

Although the transistor is a very compact amplifier and is used quite extensively in applications requiring comparatively small output power, it must take a back seat to vacuum tubes when the system requirements dictate high power—especially at the higher frequencies. A closer examination reveals why. The emitter-base n—p junction is sensitive to variations in heat. Should one small portion of the emitter-base junction area become slightly warmer than the adjacent or remaining area, the warmer area will emit more electrons per unit area than will the cooler area of the junction. As it emits a proportionally larger number of electrons, it does more work and becomes hotter than the remaining area. As it becomes hotter, it emits a still greater proportion of electrons and a run away condition rapidly develops causing the semiconductor to burn out. Thus the total input current must be limited by the input resistor (R_{in} in Figure 11) to avoid failure—even though the remainder of the transistor could easily be designed to handle a substantially higher current and operate at higher temperatures. A second transistor failure mechanism is closely related to that of thermal run away, and like the latter is catastrophic. It is known as secondary voltage breakdown. Thus by elimination the emitter from a transistor, the electron gun injected electron charge density can be kept very uniform and completely independent of the semiconductor temperature or temperature gradient. As such, the major cause of high power transistor failure can be eliminated while maintaining the high current handling capability of the collector.

When a transistor is used at higher frequencies, the rate at which the carriers diffuse across the base region becomes an inherent limitation to operation at higher powers and frequencies. In contrast, the GEISHA device injects electrons into the collector region by impact penetration through the analogous base region. Thus, much higher power at much higher frequencies are theoretically possible.

Summary

An analysis of high power vacuum tubes lead to built-in wear-out mechanisms which are attributed to the high current requirements demanded of the cathode. Reliability of high power transistors is primarily dependent on the prevention of thermal run away attributed to thermal inhomogeneities in the emitter-base junction. In this regard the GEISHA promises to eliminate the failure prone emitter and replace it with an electron gun whose cathode is required to emit but 1/4000

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of the current required by its all vacuum tube counterpart. Thus the GEISHA promises to eliminate not only the built-in wear-out and catastrophic failure mechanisms of high power vacuum tubes and transistors respectively, but to retain the best features of both technologies.

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1. U.S. Patent 2,691,076, 5 Oct. 54, A. R. Moore, *et. al.*
2. "Electron Beam Switched P–N Junctions," IEEE Transactions on Electron Device Jan. 1963, pages 8-12 by A. V. Brown.
3. "Electron Beam–PN Junction Active Devices and Measurements," Stanford Electronics Laboratories Technical Report SU-SEL-66-118, March 1967.

RADM C. O. Holmquist, USN, Chief of Naval Research, announced that this year's winner of the Research Reserve Award for General Excellence was NRRC 9-1, Chicago, Illinois, commanded by CDR John Dvorscek, USNR. The award is given to the most outstanding Research Reserve Company out of a total of 87. The Commandants of the 10 Naval Districts each choose one company from his district; from these 10 choices the panel picks a winner. The other companies chosen were:

1-2 Providence, Rhode Island	6-6 Chapel Hill, North Carolina
3-16 New Haven, Connecticut	8-3 College Station, Texas
4-4 State College, Pennsylvania	11-7 Tucson, Arizona
5-9 NRL, Washington, D.C.	12-3 Sunnyvale, California

13-5 Corvallis, Oregon

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November is the 75th birthday of x-ray. Over the years the Navy has been a leader in x-ray research. In the future the Navy will continue to use its unique capabilities for x-ray research to get closer to the full potential of x-rays.

Gun Electron Injection for Semiconductor Hybrid Amplification (GEISHA).....MAX N. YODER 42

ONR hopes through GEISHA to eliminate not only the built-in wear-out and catastrophic failure mechanisms of high power vacuum tubes and transistors, but to retain the best features of both technologies.

Four of the major areas of x-ray research and application discussed in the feature article are represented on the cover by the data used and objects studied in each. Clockwise from upper left (a) radiographs for medical diagnosis, (b) x-ray diffraction pattern for the analysis of the molecular structure of Dianin's Compound, (c) x-ray spectra for the determination of electronic band structure in TiNi, and (d) intensity scans for location of cosmic x-ray sources such as the galaxy M-87. (Photographs courtesy of: V. E. Schultz; J. and I. L. Karle and J. Flippen; D. Papaconstantopolous and C. Gilmore; and H. Friedman.)

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